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Air Pollution Effects on Forests in the ECE- region
Network Integrated Monitoring of Forest Ecosystems
Administratie Milieu Natuur en Landinrichting. Dienst Waters en Bossen,
bestuur Natuurbehoud en Ontwikkeling

**Report on the intercalibration
exercise of methods for
forest soil analysis**

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in cooperation with the Flemish
Soil expert group

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2.1. INVOLVED LABORATORIES

AUSTRIA - 9	Forstliche Bundesversuchsanstalt
BELGIUM - 15	Bodemkundige Dienst van België
CHECHOSLOVAKIA - 10	Forestry and Game Management Research Institute
DENMARK - 5	Royal Veterinary and Agricultural University
FRANCE - 7	Institut National de la Recherche Agronomique, Laboratoire d'Analyses des sols
FINLAND - 14	The Finnish Forest Research Institute, Department of Forest Ecology
Fürstentum LIECHTENSTEIN - 12	Forestry Department, Landesforstamt
GERMANY - 16	Niedersächsische Forstliche Versuchsanstalt
GREECE - 2	Ministry of Agriculture, Forest Research Institute
HUNGARY - 12	Forest Management Planning Service
IRELAND - 6	COILLTE, The Irish Forest Board
ITALY - 19	Land- und Forstwirtschaftliches Versuchszentrum
KROATIA - 1	Sumarski Fakultet, Forest Research Institute
LITHUANIA - 20	The Lithuanian Forest Research Institute
NETHERLANDS - 13	The Winand Starring Centre
NORWAY - 3	Dept. of Chemical Laboratories, Norwegian Forest Research Institute
ROMANIA - 8	Institutul de Cercetari si Amenajari Silvice
SLOVENIJA - 11	Institut za Gozdno in Lesno Gospodarstvo
SPAIN - 22	Instituto Nacional de Investigacion y Tecnologia Agraria y Alimentaria
SWEDEN - 17	Dept. of Ecology and Environmental Research
SWITZERLAND - 4	Swiss Federal Institute for Forest, Snow and Landscape Research
UNITED KINGDOM - 21	The Forest Authority, Research Division

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14. Exchangeable Ca	256
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16. Exchangeable K	294
17. Exchangeable Na	313
18. CEC	330
19. Base saturation	350
20. Total N	368
21. P	394
22. K	417
23. Ca	440
24. Mg	463
25. Na	484

PART I : DATA

Introduction

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In the context of the monitoring of forest ecosystems in the UNEP-UN/ECE-ICP forests programme, a set of soil parameters is being collected by most European countries (both EC and non-EC members). All these measurements will be compiled into a common database, so that the forest-soil condition can be evaluated on a European scale. To have a common reference for the soil analysis results, measured by different countries, the 3th Soil Expert Panel (Brussels, 1991) decided to proceed at an intercalibration exercise. This study should assess the amount of variation introduced by differing methodologies and establish the necessary need for harmonization for a correct interpretation of the ICP-forest programme soil data.

With this purpose 22 nationally designated laboratories received 4 prepared soil samples, namely 2 acidic mineral soil samples (B1 and B2), 1 calcareous mineral soil sample (A1) and 1 sample of an organic layer of a calcareous soil (A2). The guidelines for the reporting procedure and the reporting forms were provided together with the manual of the recommended reference method descriptions for the mandatory parameters.

From november 1991 until april 15 1992, the data have been collected at the secretariate of the Panel, held by the forest service of the Flemish Community of Belgium. After research of the "raw" results by the participating laboratories (june 1992) and a call for additional information (july 1992), the data collection was closed in august 1992.

Part I of this document gives an overview of the overall collected data. It informs about (i) the participants in this intercalibration run, (ii) the parameters that have been measured and (iii) the methods that are used by all these different laboratories to analyse a certain parameter.

In Part II descriptive statistics per parameter are computed and hypotheses regarding differences between methods are tested using analysis of variance and comparison of mean values.

This study aims to be a an objective basis for discussion by the Panel about comparability of methods. Therefore the authors had no intention to draw decisive conclusions.

The Soil Expert Panel should clarify following questions:

- Which methods are different from each other or which methods differ from the reference method?
- How should the results be interpreted if no reference method is prescribed?
- Which conclusions will be drawn when the variability within the reference method is large?

1.1. Participating laboratories

The results of this study are confidential. Therefore, all participating laboratories received a personal labcode. But it may be interesting to point out which laboratories participated in this intercalibration exercise. Table I lists the names and addresses in an alphabetic order, which is completely different from the numerical order of the labcodes.

TABLE I : Participating laboratories

AUSTRIA	Forstliche Bundesversuchsanstalt
BELGIUM	Bodemkundige Dienst van België
CHECHOSLOVAKIA	Forestry and Game Management Research Institute
DENMARK	Royal Veterinary and Agricultural University
FRANCE	Institut National de la Recherche Agronomique, Laboratoire d'Analyses des sols
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The Soil Expert Panel proposed of list of 36 parameters that are supposed to characterize the effects of air-pollution on the soil condition. Some of them have the status of mandatory parameters, others are optional. For the analysis of the mandatory parameters, the Panel proposes a reference method. In other cases, each laboratory is free to choose its proper method. Table II gives a list of the parameters that are included in this study.

TABLE II : List of parameters

1. pH (CaCl ₂)	19. Base saturation
2. pH (H ₂ O)	20. total N
3. Organic C	21. total P
4. CaCO ₃	22. total K
5. Electrical conductivity	23. total Ca
6. clay	24. total Mg
7. silt	25. total Na
8. sand	26. total S
9. exchangeable acidity	27. total Al
10. exchangeable Al	28. total Fe
11. exchangeable Fe	29. total Zn
12. exchangeable Mn	30. total Cu
13. exchangeable H	31. total Pb
14. exchangeable Ca	32. total Mn
15. exchangeable Mg	33. total Cr
16. exchangeable K	34. total Ni
17. exchangeable Na	35. total Cd
18. Cation Exchange Capacity	36. total Hg

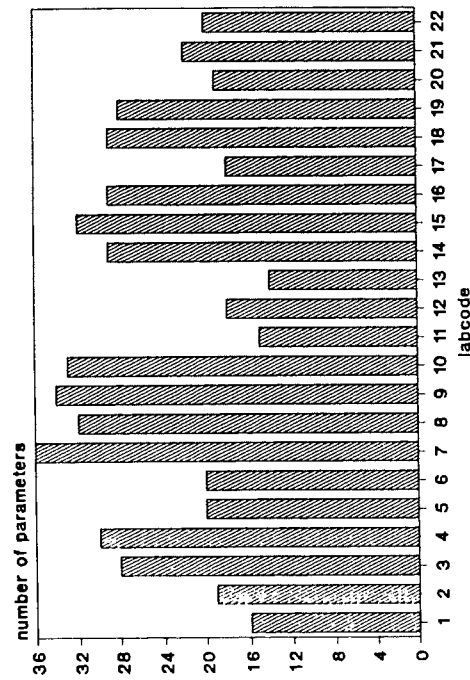
1.3. Soil samples

The soil samples that are distributed among the participating countries have various origins. The samples A1 and A2 are from Austrian soils, B1 and B2 are from Belgian soils. Labcode 14 repeated most of the A2 measurements on an extra A3 soil sample (A3 being a finer grained version of the A2). The results are included in this study, however no comparisons with A2 were made.

1.4. Response by the laboratories

Figure 1 indicates the total number of parameters that are measured by each laboratory. 50% of the participating laboratories reported about 20 different analysis. While the rest, provided reports on more than 28 parameters.

Figure 1: Reported parameters per laboratory



1.5. Method descriptions

All kinds of methods to perform a certain analysis have been used by laboratories from the various countries. For each individual parameter, an effort is made to collect the different methods involved and to group the laboratories according to equal or nearly similar methods. This list is not a final one. Some groups may be subdivided, while others will be regrouped for further analysis. Before starting the discussions on the parameter results, it is advised to evaluate these groupings before.

1. pH(CaCl₂)

- 1 Reference. UNEP - UN/ECE METHOD 9103SA.
The pH of the soil is potentiometrically measured in the supernatant suspension of a 1:5 soil:liquid (CaCl₂ 0.01M) mixture. For H and O horizons, use a 1:20 soil:liquid fraction. Take 20 g sample into a 200 ml bottle. Add 100 g CaCl₂ solution and cap the bottle. Shake for 2 hours. Calibrate the pH meter with buffer solutions (pH 7 and pH 4). Before opening the bottles for measurement, shake by hand. Immerse electrode(s) in upper part of the suspension. Read pH when reading has stabilised.
- 2 Take 30 g mineral soil and 30 ml CaCl₂ (1:1 V:V) or 10 g of O-humus and 50 ml CaCl₂ (1:5 V:V). Stir for 1 minute with a glass rod. Allow to stay for 1 hour. Stir and measure the pH in a standardised pH meter.
- 3 AFNOR X31-103. Measurement of pH value.
- 4 Measure 10 ml of air dried, <2mm soil into a labelled 50 ml beaker. Add 25 ml of CaCl₂ by pipette, stir and allow to stand for at least 30 minutes. Measure the pH using the 'Radiometer' pH meter.
- 5 1 g organic soil or 5 g mineral soil is mixed with 25 ml liquid. Shake by hand and stand for 16 hours. Shake by hand, wait for sedimentation and measure in the supernatant part.
- 6 0.01M CaCl₂.
- 7 Mix 20 g soil and 100 ml KCl (1:5 V:V).
- 8 1N KCl.
- 9 The potentiometric pH-measurement is carried out by mixing the sample with KCl in the ratio of 1:2.5 (V:V). 15 ml sample is mixed with 37.5 ml KCl 1M in a 100 ml beaker glass. The mixture has to be stirred about 5 times during 4 hours. After 4 hours the pH is measured in the suspension with a calibrated pH meter.

2. pH (H₂O)

- 1 UNEP - UN/ECE METHOD 9103SA.
The pH of the soil is potentiometrically measured in the supernatant suspension of a 1:5 soil:liquid mixture. For H and O horizons, use a 1:20 soil:liquid fraction. Take 20 g sample into a 200 ml bottle. Add 100 g DI-water and cap the bottle. Shake for 2 hours. Calibrate the pH meter with buffer solutions (pH 7 and pH 4). Before opening the bottles for measurement, shake by hand. Immerse electrode(s) in upper part of the suspension. Read pH when reading has stabilised.
- 2 Take 30 g mineral soil and 30 ml H₂O (1:1 V:V) or 10 g of O-humus and 50 ml H₂O (1:5 V:V). Stir for 1 minute with a glass rod. Allow to stay for 1 hour. Stir and measure the pH in a standardised pH meter.
- 3 AFNOR X31-103. Measurement of pH value.
- 4 The pH of the soil is potentiometric measured in the supernatant suspension of a 1:2 soil:liquid (H₂O) volume/volume mixture for both mineral and organic soils.
Procedure: each soil sample is completed in duplicate for pH. Using 10 ml scoop, place 10 ml of air dried soil (<2mm) into a 50 ml beaker. Add 20 ml distilled water with a dispenser. With a stirring rod, stir soil/water vigorously. Cover. Allow to stand for minimum 2 hours or preferably overnight. Calibrate the pH meter with buffer solutions (pH 7 and pH 4). Check with intermediate buffer for accuracy. Immerse electrode(s) in upper part of suspension. Read pH when reading has stabilised.
- 5 10 g sample + 25 ml.
- 6 1 g organic soil or 5 g mineral soil is mixed with 25 ml liquid. Shake by hand and stand for 16 hours. Shake by hand, wait for sedimentation and measure in the supernatant part.
- 7 unspecified.

temperature 125°C for 45 min. Then allow to cool and make to volume with DI-water and swirl the flask. After sedimentation of soil particles, pipette 25 ml of solution into suitable vessel for titration. Add 2 ml (for O-horizons 3 ml) KI 20% solution and titrate with 0.2N Na₂S₂O₃ solution. After disappearing of brown colour, add 2 ml starch solution (1 g starch and 250 ml hot water). A violet colour appears. Titrate again with 0.2N Na₂S₂O₃ until the violet colour changes into a green one.

Calculation:

- for mineral soils: $C.Org(\%) = 0.6 * f * (a-b)$

- for O-horizons: $C.Org(\%) = 3 * f * (a-b)$

where f = factor of 0.2N Na₂S₂O₃

a = amount of 0.2N Na₂S₂O₃ for blank

b = amount of 0.2N Na₂S₂O₃ for sample

Soil organic matter content by ignition loss.

5 g soil sample is weighed on an analytical balance and put into an annealing pot of known volumen. The soil sample will be dried for 4-10 hours in drying oven on 105°C temperature, depending on the soil texture. It is then left in exsiccator to cool to room temperature. Weigh again on an analytical balance. Then it is put into an ignition oven and kept there for 3 hours at a temperature of 700°C. Then it is cooled to 100°C and put into exsiccator. Its weight is then determined on analytical balance.

Evaluation: in case of carbonate-free soil sample, the humus content of air dry sample (g/100 g soil) is:

$$H\% = [(f-a)/(f-b)] * (100 - \text{soil moisture in air dry condition})$$

If the soil sample contains carbonate and the amount is known, the ignition loss can be corrected:

$$H\% = [(f-a)/(f-b)] * (100 - \text{soil moisture in air dry condition}) - w(\text{CaCO}_3) * 0.44$$

$$C = H\% / 1.72$$

Thermal combustion with oxygen. Separation of the combustion gasses with separation columns and detection in a heat conductivity cell.

7-10 mg of the humus sample is weighed into tin containers which are closed with a special tweeze. Within 40 samples 8 standards (acetanilide) are measured. The measurement is carried out with an automatic sampler. For soil samples: 45-50 mg are filled into tin containers.

Titration.

A blank digestion is included with each set of

7

3. Organic Carbon (%)

1 Reference. UNEP -UN/ECE METHOD 9104SA.

Dry combustion.

2 Wet ashing.

3 labcode 2: Wet Oxidation (K₂Cr₂O₇ + H₂SO₄)

(Walkley, 1946).

labcode 8: Walkley & Black.

labcode 15: Walkley & Black. Oxidation of the O.M. by

K₂Cr₂O₇ in the presence of conc. H₂SO₄. The excess

K₂Cr₂O₇ is titrated by FeSO₄ with difenylamine

indicator. Use 0.6 g of soil. Calculation: humus = C *

1.72.

Organic matter % loss on ignition.

The organic matter content of the organic layer is assessed gravimetrically by dry combustion of the organic matter in a furnace at 850°C. The loss in weight gives an indication of the content of the organic matter of the sample.

Procedure: Heat a porcelain crucible (bottom area of at least 10 cm²) with its cover for 1.5 hours in a furnace at 850°C. Cool in the open to about 150°C. Put in a dessicator, cool to room temperature and weigh it together with its cover. Then weigh precisely 2.5 g of an air dry soil sample into the crucible ("A" gram of sample). Put the crucible and its cover separately in the furnace and raise the temperature gradually (during 1.5 hours) from room temperature to 850°C. Maintain this temperature during 2.5 hours. Then cool off in the open, the crucible being covered, to about 150°C and put the crucible in a dessicator until the sample has cooled off to room temperature (45 min.) then weigh the crucible, and calculate the weight of the sample ("B" gram).

Calculation: the weight of the air dry sample "A" has first to be corrected for the moisture content (W, in %)

(A' = weight of oven dry sample):

$$A' = [A / (100 + W)] * 100$$

$$O.M. (in \%) = [(A' - B) / A'] * 100$$

5 AFNOR X31-109. Determination of Org.C by sulfochromic oxydation.

6 Tjurin modified method.

Principle: Wet combustion of organic material with a mixture of K₂Cr₂O₇ and H₂SO₄. After reaction the excess of dichromate is determined by iodometric titration.

Procedure: Weigh 0.4 g of soil sample (for O-horizons 0.2 g), gently ground, into a 100 ml (for O-horizons 250 ml) volumetric flask. Add 45 ml (for O-horizons 75 ml) mixture solution (40 g dichromate dissolved in 900 ml H₂O in 2000 ml volumetric flask, then add carefully 1000 ml H₂SO₄, stir and cool, after cooling complete volume to 2000 ml). Place flask into heating oven at

9

determinations. This is produced in the same way as the following but no sample is added.
Accurately weigh (to 0.0001 g) a sample of air dried, <2mm soil into a labeled round bottomed flask. The amount used should be decided by comparing the colour of the soil with the guide samples based on the colour of soils of known organic carbon content. You should not use more than 0.5 g of sample. Add approx. five anti-bumping granules to each flask. To each flask add 20 ml +/- 0.1 ml of potassium dichromate solution by Zipette, followed by 30 ml +/- 0.1 ml of acid mixture also by Zipette. These reagents must be added in this order, and separate Zipettes must be used. Heat the mixture rapidly to boiling under reflux, and keep boiling for at least 20 minutes, but not more than 25 minutes. Allow the flasks to cool, and wash down the inside of the condenser with demineralised water into the flask. Titrate the digested mixture with the ammonium ferrous sulphate solution, using 4-5 drops of the prepared indicator. The end point is as the purple colour changes to green. Record the titre, which should be at least 10 ml; if not, repeat using less soil as appropriate.

Calculation:
- A = titre of blank / 20
 1.545 * (blank titre-sample titre)
- Org.C = $\frac{\text{sample mass(g)} * 10}{\text{sample mass(g)} * 10}$

Report on oven-dry basis.

LECO-C

unspecified

4. CaCO₃ (%)

- 1 Reference. UNEP - UN/ECE METHOD 9102SA.
AFNOR X31-105. Method suitable for H- and O-horizons and for mineral horizons if pH(CaCl₂) > 6.
Principle: the soil sample is treated with a strong acid. The loss of CO₂ gas is measured volumetrically.
Procedure:
1. All the material is milled to pass a sieve of 0.2 mm.
2. Take a sample amount according to table 1.

Table 1

CaCO ₃ content in % predicted	sample amount ml in grammes
0.5-3.9	10
4 -9.9	5
10 -19.9	2.5
30 -39.9	1
40 -79.9	0.5
+80	0.25

3. Bring the sample in the reaction vessel, place the tube with 10 ml of hydrochloric acid (1/1) in the vessel and close the system. Before closing the 3-way valve, make sure that the zero reading is corrected. Mix the hydrochloric acid with the sample by shaking the reaction vessel. Follow and equilibrate permanently the height of the water column with the ampoul. (E). Read the volume (V1) when no further change occurs in the liquid level.
Under the same conditions of temperature and pressure, proceed like under n°3, with 0.050 g, 0.100 g, 0.150 g and 0.200 g of calcium carbonate. Determine the calibration curve and with this curve and V1 the amount m2 of CaCO₃ in the sample.

Calculation: % CaCO₃ = $100 * \frac{m2}{m1}$
m1 = sample amount in grammes

m2 = amount of CaCO₃ in grammes in the sample.

labcode 2: Calcimeter (Allison & Moodie, 1965)

Wet ashing.

Gravimetric determination.

4 % CaCO₃ by TNV method. Weigh accurately 0.25 g, 0.50 g and 1.00 g pure CaCO₃ into 300 ml tall form pyrex beakers. Weigh between 0.5-2 g soil sample depending on the expected CaCO₃ content. Using a safety bulb, pipette 50 ml of 0.5M HCl and add slowly down the side of beaker. Add a large watch glass and shake cautiously to dissolve sample or standard.
With difficult samples (i.e. ground limestone) place on a hot plate and bring to just boiling for approx. 5 minutes. Allow to cool. Titrate the excess HCl with 0.5M NaOH using 3-5 drops of phenolphthalein as indicator. Record the titration figure.

Calculations:

1. 50.00 - titration figure (neutralising value)
2. calculate the weight of CaCO₃ - equivalent using: 1 ml 0.5M HCl = 0.025 CaCO₃
3. calculate the T.N.V. (% CaCO₃) as follows:

T.N.V. = $\frac{\text{CaCO}_3\text{-equivalent in sample(g)} * 100}{\text{wt of sample (g)}}$

Apply a moisture factor if required.

5 A suitable amount of soil is weighed into a 250 ml Erlenmeyer flask and 80 ml H₂O is added. A plastic foam plug is loaded with KOH solution and placed in a special housing for CO₂ absorbent. 10 ml 9M HCl is injected to the stoppered flask and is placed on a rotary shaker for 2 hours. The plastic foam plug is transferred to another flask and 100 ml water is added for washing. 20 ml 1M BaCl₂ is added and the excess of OH⁻ is neutralized with phenolphthalein as indicator. To determine the amount of BaCO₃ the sample is titrated with methylorange as indicator. A blank is performed and subtracted from the titration.

- 6 1. Before any measurements are made the apparatus must be checked for leaks, as these are normally not visible to the eye. This is done by using 1.000 g of oven-dried calcium carbonate instead of soil in the procedure beginning at 2 (below). The result must lie between 99% and 101% for work to proceed. If it does not, repeat the measurement.
2. Accurately weigh, to 0.0001 g, a sample of air-dried, <2mm soil, into a labelled, 250 ml round-bottomed flask. The amount of soil is decided by prior testing of the soil. This is done by adding a few drops of approx. 12M HCl to approx. 10 ml of wetted soil in a petri dish. The amount of soil to use is estimated by observing the effervescence:

Very little reaction: 10 to 20 g

Slight to moderate reaction: 5 to 10 g

Moderate to vigorous reaction: 2 to 5 g

Very vigorous reaction: 1 to 2 g

The measurement should not be made with less than 1 g of soil.

3. Place a magnetic follower in the labelled flask and clamp into position as shown in Fig.1.
4. Open TAP A & TAP B. Raise the reservoir bottle so that the level in both tubes rises above zero graduation in the manometer. Close TAP B.
5. Slowly lower the reservoir bottle so that the bottom of the meniscus of the liquid in the manometer rests on the zero graduation. Close TAP A.
6. Open acid inlet TAP C, running in enough acid to make the sample just fluid. Turn on the magnetic stirrer. Leave for several minutes.
7. When reaction appears to have ceased, open TAP B and allow liquid in capillary tube A to become level with that in the manometer. Close TAP B. Add further acid. Repeat this procedure until the reaction ceases. NOTE: the reaction mixture should be left for no more than one hour, to avoid excessive dissolution of the carbon dioxide in the acid mixture. Some magnesium rich carbonates will continue to react for several hours, in which case this fact should be recorded against the result.
8. Read the volume of gas produced. Record air temperature and pressure at the same time.

Basis of the calculation:

Need to correct volume of CO₂ produced to STP and equate to mass of CaCO₃.

STP = 760 mm Hg, 273°K and 1 g CO₃ = 224 ml CO₂

- $(\text{vol CO}_2 \cdot P_1) / T_1 = (\text{V}_2 \cdot P_2) / T_2$

- The corrected volume of CO₂ is (BP = barometer pressure):

$(\text{vol CO}_2 \cdot \text{BP} \cdot 273) / (C + 276) \cdot 760$

- Mass of CO₃:

$\text{Vol CO}_2 \cdot \text{BP} \cdot 273 / (C + 273) \cdot 760 \cdot 224$

- %CO₃ (S = sample mass):

$\text{Vol CO}_2 \cdot \text{BP} \cdot 273 \cdot 100 / (C + 273) \cdot 760 \cdot 224 \cdot S$

- $273 \cdot 100 / 760 \cdot 224 = 0.1604$

Calculation:

- % CaCO₃ equiv. : $\text{Vol CO}_2 (\text{ml}) \cdot \text{BP} (\text{mm Hg}) \cdot 0.1604 / \text{sample mass (g)} \cdot (\text{Temp} + 273)$

unspecified

5. Electrical Conductivity Ec (mS/m)

1 labcode 6: The electrical conductivity of the soil is measured in the supernatant suspension of a soil:liquid (DI-water) mixture.

Procedure: blank = 100 g distilled water. Weigh 20 g (mineral soils), 5 g (organic soils) into 250 ml PP bottle (with a suitable spoon). Add 100 g distilled water with a dispenser and cap the bottle. Shake for 2 hours at a setting of 10 on the orbital shaker (300 RPM). Allow to stand overnight. Calibrate/check meter accuracy using 0.1M KCl standard solution, ref st. = 25°C. Immerse electrode in upper part of suspension and read Ec value at 25°C when the reading has stabilised. labcode 15: potentiometrically measure the conductivity of the water extract with Ec-meter (demineralsed water, extraction ratio 1/5 or 20 g sample/100 ml H₂O).

2 AFNOR X31-113. Determination of aqueous soils extracts.

3 10 g sample, 100 ml. report: µS/m

4 cfr method 1, the watery solution is separated from the soil before Ec-measurement.

6..8. Particle size distribution: clay, silt, sand (%)

- 1 Reference. UNEP - UN/ECE METHOD 9101SA.
Separation of the mineral part of the soil into various size fractions and determination of the proportion of these fractions. The analysis includes all soil material, i.e. including gravel and coarser material, but the procedure below is applied to the fine earth (<2mm) only. Of paramount importance in this analysis is the pretreatment of the sample aimed at complete dispersion of the primary particles. Therefore, generally, cementing materials (usually of secondary origin) such as organic matter and calcium carbonate are removed. Removal of organic matter by H₂O₂ and carbonates by a mildly acid buffer pH 5 is normally carried out. After shaking with a dispersing agent, sand is separated from clay and silt with a 63 µm sieve. The sand is fractionated by dry sieving, the clay (<2µm) and silt (2-63µm) fractions are determined by the pipette method.
 - 2 The hydrometer method, without any pretreatment or removal of CaCO₃.
 - 3 AFNOR X31-107.
 - 4 Areometer method (sieving of sand fraction and hydrometer for silt and clay fractions).
 - 5 The particle size distribution is determined without destruction of carbonate and organic matter. If organic matter exceeds 5% (9% C.org) no particle size distribution is done. The sand fraction is determined by sieving. The silt and clay fractions are determined by sedimentation (pipette method) comparable to the reference method.
 - 6 unspecified.
9. Exchangeable acidity (cmol+/kg)
- 1 Reference. UNEP - UN/ECE METHOD 9106SA.
There are 2 alternatives:
1. Titration of the BaCl₂ extraction to pH 7.8
2. Sum of acid cations Al³⁺ + Fe²⁺ + Mn²⁺ + (H⁺)
- Principle: The sample is extracted by unbuffered 0.1M BaCl₂. The exchangeable acidity is determined
a) by titration of this solution to pH 7.8 or
b) by measuring Al³⁺, Fe²⁺, Mn²⁺ and (H⁺)
- Procedure: Place 2 g of soil passed over a 2 mm sieve plus 20 ml of 0.1M bariumchloride in a 30 ml plastic

centrifuge tube and shake for 2 hours. Centrifuge and retain the supernatant solution for analysis.

- a) Titration of the BaCl₂ extraction to pH 7.8
- b) Measuring the acid cations (exchangeable cations)

Al, Fe, Mn: by AAS or ICP

(H): alternative 1: by the "German" method (difference in pH before and after extraction and model)

alternative 2: by titration H = (H + Al) titration - Al (AAS or ICP). This titration is recommended to do on a 1M KCl extraction rather than 0.1M BaCl₂

The exchangeable acidity is the sum of the amounts of the acid cations Al³⁺, Fe²⁺, Mn²⁺ and (H⁺).

total CEC - Σ(Ca²⁺, Mg²⁺, K⁺). Analysis of CEC and exchange cations: see respective method descriptions.

1M NH₄NO₃.

The exchangeable acidity is determined by an absolute end point titration to pH 7.0. Blank value is corrected. This method measures contribution of all acidic cations such as H, Al, Fe etc. The results are expressed in mmol/kg dry basis.

1M KCl extract, titration up to pH 8.2

labcode 8: Le procédé d'extraction pour Al³⁺ échangeable et les ions H⁺ extractibles avec la solution d'un sel, résidu en extraction et lessivage du sol avec une solution de KCl 1N. Méthode de travail: 5 g sol et 100 cm³ KCl 1N. Après une heure on agite 30 min., puis on met en repos pour se clarifier et se décanter, la solution se passe par un filtre et on collecte la solution filtrée dans un ballon de 20 cm³. On répète l'extraction deux fois avec 50 cm³ du solution de KCl, on agite chaque fois 15 min. et on collecte l'extraire dans le même ballon. On ajoute une solution KCl 1N jusqu'à 200 cm³.

Dosage d'acidité d'échange (Al³⁺ + H⁺): 100 ml de l'extraire se chauffe jusqu'à l'ébullition pur éliminer le CO₂ et on titre à chaude avec une solution de NaOH 0.01N dans la présence de fenolftaleine.

Calcule: (Al³⁺ + H⁺) meq/100g = a*f*n*v*100 / v*m

a = cm³ NaOH 0.01N utilisé pour la titration
f = facteur de correction pour la solution NaOH 0.01N

v = volume totale de l'extraire (200 cm³)

n = concentration de la solution NaOH (0.01N)

v = volume de l' échantillon titré (100 cm³)

m = quantité de sol (5 g)

Dosage d' Al^{3+} échangeable: dans l'extrait utilisé pour déterminer l'acidité totale s'ajoute 10 cm³ de solution NaF et on titre avec une solution de HCl 0.01N jusqu'à la disparition de la couleur rose qui ne revient pas dans 2 min.

Calcule: $Al^{3+} \text{ meq/100g} = a * f * n * v * 100 / v * m$

a = cm³ HCl 0.01N utilisée pour la titration
f = facteur de correction de la solution HCl 0.01N

n = concentration de la solution HCl 0.01N

v = volume d'extrait totale

v = volume de l'échantillon titré

m = quantité de sol (5 g)

Calcule de H^+ : $H^+ \text{ meq/100g} = (Al^{3+} + H^+) - Al^{+3}$
labcode 12: The exchangeable acidity is the soil acidity that can be titrated and measured after having been shaken with a neutrally dissoluble KCl solution of 1 mol/l concentration. 40 g of soil sample is measured on a tare balance and then put into a shaking flask. Then 100 ml KCl 1M is added with a pipette, the shaking flask is then closed and the soil suspension will be shaken in a rotary shaking machine for 1 hour. Then it will be filtered into a beaker through filter paper. 50 ml of the filtrate will be titrated to pale rose-red colour with NaOH 0.1M in the presence of phenolphthaleine indicator.

$$y^2 = v * r(NaOH) * 2.5$$

V: ml NaOH

r: reduction factor

labcode 18: Weigh 100 g soil in perkolation column, add 40 ml BaCl₂ 0.1M twice. Perculate after 30 min., rinse the column twice with 50 ml DI-water. Dilute with water and make to volume mark. (use 250 ml flask). Pipette 200 ml eluat in a bottle, add 4-5 drops phenolphthaleine.

Titrate with 0.1M NaOH until a light rose colour appears. Repeat this procedure with a blank.

Determination of the exchangeable Al^{3+} cations:

add 10 ml 1M KF solution and titrate with 0.1M HCl until discoloration. Wait 30 minutes, titrate, if necessary, with HCl 0.1M to obtain a clear titration-endpoint.

Calculation: expressed in mmol/z/100g soil

$$(ml \text{ NaOH} - ml \text{ NaOH blank}) * 0.1 * 100 * 1.25$$

exch.acidity =

2.5

$$exch.Al = \frac{(ml \text{ HCl sample} - ml \text{ HCl blank}) * 0.1 * 100 * 1.25}{2.5}$$

2.5

exch.H = BaCl₂-acidity - BaCl₂-exch.Al

Σ acid cations = CEC - Σ basic cations
(determination method: see resp. method descriptions)

H^+ . 15 ml air dried sample (exact weight recorded) + 150 ml 0.1M BaCl₂ solution. Left to stand overnight then shaken on an end-over-end rotating shaker for 1 hour. Filtered through a Schleicher & Schuell 5893 Blauband filter paper. Titration of 50 ml of the filtrate with 0.05N NaOH to endpoint pH 7.00. Amount of 0.05 N NaOH titrated corrected for blank (50 ml of 0.1M BaCl₂ solution).

Σ acid cations. Detection of cations in NH₄Cl extract.

20 g mineral soil and 100 ml 1M KCl is shaken for 2 hours. The suspension is filtrated and 50 ml is pipetted for titration to pH 8.2 with 0.1M NaOH. 10 ml 4% NaF is added and titrated with 0.1M HCl to pH 8.2.

Titration to pH 7.8 from a soil extract with 1M KCl.

Extraction with cobaltihexamine (50 mmol+/l) and titrimetric determination of Al and H^+ . Ratio 2.5 g/50 ml soil:solution.

Reagents: Distilled water through which a stream of CO₂-free air bubbles has been passed overnight to decrease the dissolved CO₂ content.

0.5M Ba(COOCH₃)₂. Adjust to pH 7 using a little dilute NH₃ or CH₃COOH solution as appropriate.
0.025M Ba(OH)₂ standardized. Dissolve 14 g Ba(OH)₂ and 2 g BaCl₂ in 900 ml of hot CO₂-free distilled water. Cool and make to 1 litre. Transfer to a burette and reservoir protected against CO₂ and leave to settle overnight.

Immediately before use standardize the solution against 0.01M HCl, which in turn is standardized against 0.05M Na₂CO₃ solution.

Procedure: Prepare leaching tubes as for exchangeable bases. Weigh 10 g <2mm air-dry soil and mix with an equal amount of coarse (2-4 mm) silica grains (not acid washed). Transfer the mixture onto the filter pad in the leaching tube. Thoroughly moisten the mixture in the tube with neutral Ba(COOCH₃)₂ solution, taking care not to add so much that excess drains from the tube. Then invert a 250 ml volumetric flask of the same solution into the tube. Control the rate of leaching to about 15 ml/hour by means of a pinchcock on the outlet from the leaching tube. Titrate all the leachate to pH 7 with standardized 0.025M Ba(OH)₂, using a calibrated pH meter whilst stirring rapidly. Record titre (T). Make blank titre determination (B).

Calculation:

extractable acidity = 20M(T-B)

unit: me/100 g air-dry soil

where M = molarity of the Ba(OH)₂ solution

Report on an oven-dry basis.

unspecified

10..17. Exchangeable cations (cmol+/kg)

- 1 Reference method. UNEP - UN/ECE METHOD 9106SA.
The sample is extracted by unbuffered 0.1M BaCl₂ and in this solution the exchangeable cations are measured.
Place 2 g of soil passed over a 2 mm sieve plus 20 ml of 0.1M bariumchloride in a 30 ml plastic centrifuge tube and shake for 2 hours.
Centrifuge and retain the supernatant solution for analysis.
a) Titration of the BaCl₂ extraction to pH 7.8
b) Measuring the acid cations (exchangeable cations)
Al, Fe, Mn: by AAS or ICP
(H): alternative 1: by the "German" method (difference in pH before and after extraction and model)
alternative 2: by titration $H = (H + Al)$ titration - Al (AAS or ICP). This titration is recommended to do on a 1M KCl extraction rather than 0.1M BaCl₂

2 Ca²⁺, Mg²⁺ and K⁺: 10 g of sample + 100 ml 1N NH₄Ac pH 7 in a 300 ml flask. Shake in a mechanical shaker (horizontal) for 1 hour. Filter through paper (Whatman No 42). Measure concentration of cations in AAS (Ca and Mg) and F.E. (K).
Al³⁺: sample extraction with 1N KCl. Colorimetric determination of Al³⁺ concentration in the extract with the "aluminium" method (Chenery, 1948).

3 1M NH₄NO₃. The exchangeable cations are analysed in the NH₄NO₃ extract by ICP-AES. The results are reported either μmol or mmol/kg dry basis.

4 labcode 4: 1M NH₄Cl * 5 g in 50 ml * 1 hour shaking; ratio 10 g/100 ml
labcode 10: The soil sample is extracted with unbuffered ammoniumchloride solution of sufficient strength to remove the exchangeable cations. This cations are determined by using AAS (flame method). Procedure: place 30 g of soil sample into a 250 ml PE bottle. Add 150 ml 1M ammoniumchloride solution and cap the bottle. Shake for 1 hour on the end-over-end shaking machine. Then overnight and shake again 1 hour the following day. Then filtrate through the filter paper and measure the exchangeable cations by AAS (method of calibration curve) according to the manual.

5 AFNOR X31-130. Determination of cationic exchange capacity and exchangeable cations, method n°2: cobaltihexamine, ratio 5 g / 100 ml.

- 6 Cations are exchanged out of the soil with an exchanging solution of 0.2N BaCl₂, which has been buffered to pH 8.1. On course of the process Ba-ions will be fixed to the soil. The exchanging solution has to have a value of pH 8.1.
2.5 g previously sifted (0.25 mm sieve) soil sample is put into a pot of 150 ml, suitable for vibration, then 50 ml of a 0.2N BaCl₂ solution of pH 8.1 is added to it and it will be shaken for 2 hours. After having been shaken the suspension is filtered through a MN Co blue (640 DD) filter paper into a 200 ml measuring flask. The soil on the filter is first rewashed with 12.5 ml BaCl₂ 0.2N, then with carbonic-acid free distilled water, with which the flask is filled to the mark.

7 The procedure is based on extraction of a soil sample at room temperature with a 0.01M solution of silver thiourea (AgTU). The affinity of AgTU for the negatively charged soil particles is sufficiently high to account for a complete saturation by AgTU, even when the soil contains relatively high concentrations of other salts. Weigh out 0.25 - 20 g of soil depending upon the soil type and method, as shown below, and transfer it to a polyethylene bottle of 100 ml. Add 50.0 ml of the 0.01M AgTU solution and close the tubes tightly. Prepare also a blank. Shake for 4 hours. Filtrate the soil. This solution is used for the Al, Fe, Na, K, Ca and Mg determinations.

8 20 g mineral soil and 100 ml 1M KCl is shaken for 2 hours. The suspension is filtrated and 50 ml is pipetted for titration to pH 8.2 with 0.1M NaOH. 10 ml 4% NaF is added and titrated with 0.1M HCl to pH 8.2. Mn, Mg and Ca are determined by AAS. K is determined on an extraction with 1M NH₄Cl by flame photometry.

9 Extraction avec une solution de CH₃COONH₄ 1N au pH=7. Pour déplacer quantitativement les cations échangeables dans la solution, il faut éliminer en avance les carbonates (CaCO₃, Na₂CO₃, MgCO₃), qui sont partiellement solubles dans la solution extractante. Les carbonates sont décomposés avec une solution de CH₃COONH₄ 1N, jusqu'à l'absence d'effervescence. Méthode de travail: 10 g sol blutée par un sas de 2mm. On ajoute 100 cm³ CH₃COONH₄ 1N pH 7, on agite 15 min. On met en repos pendant 24 heures et on filtre. Le sol resté sur le filtre se lave avec CH₃COONH₄. On continue le lavage jusqu'à ce que les ions de Ca²⁺ sont lessivés (on vérifie avec oxalate d'ammonium 4%). On passe dans un ballon de 500 ml. L'extraît s'évapore sur un bain de sable jusqu'au volume d'approximativement 20 cm³. On passe quantitativement dans des capsules de quartz et on rince les verres avec de l'eau chaude. Le contenu de la capsule s'évapore au sec sur un bain de sable et on calcine le résidu obtenu à l'approx. 400°C jusqu'à le résidu prend la couleur blanche. Après le

18. Cation Exchange Capacity (cmol+/kg)

- 1 Reference. UNEP - UN/ECE METHOD 9106SA.
The CEC is calculated by making the sum of all exchangeable cations. Depending on the applied analytical method, there are 2 alternatives to calculate the CEC:
1. Sum base cations + titratable exchangeable acidity
2. Sum base cations + sum acid cations
 - 2 Saturation of sample with 1N Na₂Ac at pH 8.3. Wash excess Na₂Ac with ethyl alcohol. Replace Na⁺ with NH₄⁺Ac at pH 7.
 - 3 1M NH₄NO₃. $\Sigma(H^+, Na^+, K^+, Ca^{2+}, Mg^{2+})$, where $H^+ =$ exchangeable acidity and is obtained by titration. The results are expressed in terms of mmol(c)/kg dry basis.
 - 4 labcode 4: $\Sigma(Na, K, Ca, Mg, Al, Fe, Mn, H)$ (NH₄Cl-extract). H is obtained from the KCl-extract after masking Al.
 - 5 labcode 16: The soil is percolated with 1N NH₄Cl solution in a column during 5 hours. The cations at the soil surface are exchanged by the ammonium cations. Procedure: 2.5 g of the soil sample is filled into a column and soaked with some ml 1N NH₄Cl solution overnight. The next morning 100 ml of the 1N NH₄Cl solution is percolated through the column. The percolation is regulated by pump, so that the procedure at least needs 4 hours. The effective CEC is the sum of all exchangeable cations (Na, K, Ca, Mg, Fe, Al, Mn, H).
 - 6 AFNOR X31-130. Determination of cationic exchange capacity and exchangeable cations, method n°2: cobaltihexamine, ratio 5 g / 100 ml.
 - 7 Back-exchange with 0.2 mol/l HCl.
- The procedure is based on extraction of a soil sample at room temperature with a 0.01M solution of silver thiocrea (AgTU). The affinity of AgTU for the negatively charged soil particles is sufficiently high to account for a complete saturation by AgTU, even when the soil contains relatively high concentrations of other salts. Weigh out 0.25 - 20 g of soil depending upon the soil type and method, as shown below, and transfer it to a polyethylene bottle of 100 ml. Add 50.0 ml of the 0.01M AgTU solution and close the tubes tightly. Prepare also a blank. Shake for 4 hours. Filtrate the soil. Pipette 1.0 ml of this solution into a 100 ml volumetric flask and dilute to ca. 50 ml with water. Add then 10 ml 1M HNO₃ in the flask and fill up to the mark with water. Mix well. Measure the concentration of Ag in the standard series, the samples and the blank by ICP at a wavelength of 328.07 nm. Calculations:

réfroidissement des capsules, le résidu se traite avec 25 cm³ HCl 10% et s'évapore au sec. On répète l'opération avec HCl 10%. Le contenu des capsules s'évapore encore une fois au sec, on le met en repos pendant 1 heure au chaude et puis on recommence avec 2 cm³ HCl 10% et un peu de l'eau distillée bouillante. On filtre dans des ballons de 100 cm³ et on rince les capsules et les papiers filtrants avec des petits portions de l'eau distillée bouillante jusqu'à ce que dans le filtrat il ne s'identifie plus des ions Cl⁻ (on vérifie avec une solution de AgNO₃ 1.7%). Après le lavage complet des ions de Cl⁻ les ballons se remplissent au signe avec de l'eau distillée. Dans la solution obtenu on détermine: Ca, Mg, Na et K.

- 10 0.1M CaCl₂, ratio 2 g / 20 ml
- 11 Transfer 5 g +/- 0.01 g of air-dried, <2mm soil, to a labelled 50 ml borosilicate glass beaker. Add 20 ml +/- 2 ml of 1M ammonium acetate solution, stir well with glass rod, cover with a watch glass and allow to stand overnight.
Filter the contents of the beaker through a Whatman n°44 filter into a labelled 250 ml volumetric flask.
Leach the soil on the filter paper with successive 25 ml volumes of 1M ammonium acetate solution, collecting all the leachates in the flask containing the original filtrate. Continue leaching until nearly 250 ml have been collected in the volumetric flask. Discard the soil and filter paper, and fill the flask to the mark with 1M ammonium acetate solution. Retain this solution for the determination of calcium, magnesium, sodium and potassium.
Carry out a blank determination in each batch in an identical manner, but omitting the soil.
Determination of calcium, magnesium, sodium and potassium by AAS. Use resp. calcium, magnesium, sodium and potassium working standard solutions (resp. RSPUR62, RSPUR39 RSPUR63, RSPUR60). Set the machine according to the instruction handbook (SSHQ/SOPE1). Prepare a calibration curve using the standards prepared above.
Pipette an aliquot (v ml, but not exceeding 10 ml) of the soil extract into a 25 ml volumetric flask. Dilute to 25 ml with demineralised water (SSHQ/SOPE2). Measure the concentration of Ca, Mg, Na and K in the sample extracts. Calculation:
A = concentration of Ca (Mg, Na or K) in sample
B = concentration of Ca (Mg, Na or K) in blank
v = aliquot used (ml)
(A-B) * 6.25/v = me/100 g of soil
Report on oven dry basis.

12 unspecified

$$\text{CEC}(\text{meq/kg}) = (b-a) * 50 / w * \text{mAg}$$

- a = concentration of Ag measured in the samples (mg L⁻¹)
 b = mean concentration of Ag measured in the blanks (mg L⁻¹)
 w = weight of oven dry soil (g)
 mAg = molar weight of Ag = 107.87 g

8 $\Sigma(\text{Ca}^{2+}, \text{Mg}^{2+}, \text{Na}^{+}, \text{K}^{+}, \text{H}^{+})$ corrected for soil moisture content. Different method of measuring the exch. cations.

9 0.1M BaCl₂ at pH 8.1, 1N

10 Determination of the effective CEC is only then interesting when the pH of the soil sample is less than 6.8, because the results at higher pH are practically identical to the potential CEC. The soil sample is saturated with Ba under soil pH. Therefore non buffered solutions are used. The Ba saturated soil sample is treated with a 1M ammoniumchloride solution. The exchanged Ba is determined by ICP-AES, flamephotometer or AAS (wavelength 553.6).

Procedure: Prepare the percolation tube and fill with 2.5 g soil mixed with 15 g quartz sand. Soak with 40 ml BaCl₂ 0.1M, and leave to stand overnight. Next morning the solution is percolated slowly. Repeat this percolation 2 times with 40 ml BaCl₂, leave 30 minutes before percolating. Rinse twice by soaking the soil sample with 50 ml water, percolate after 30 min. This solution is used to determine the exchange acidity and the Ca, Mg K and Na contents. Replace the bottle. Soak the sample again with 40 ml NH₄Cl 1M and percolate after a while. Repeat this procedure 4 times with 40 ml NH₄Cl and leave to stand for 30 min. Make to volume with NH₄Cl. Determine the Ba content by AAS, ICP-AES or flamephotometer.

Calculation for AAS:

Calibration solutions: pipette 0 (blank), 2.5, 5, 10, 15, 20 and 25 ml of Ba solution (100 ppm Ba) in 100 ml flasks. Add 10 ml Cesiumchloride solution (1500 ppm Cs) and 1M NH₄Cl (add enough to compensate for the eluat volume). Make to volume with water. Pour 1ml Eluat (V2) and 5 ml Cesiumchloride in a 50 ml flask and make to volume (V1). Measure the extinctions of the calibration solutions and the diluted eluat against the blank at 553.6 nm.

Calculate the calibration curve. Recalculate the extinctions to Ba content values.

$$[(c1-c0)V1 * 10]/(68.8 * V2) = \text{mmol/z}/100 \text{ g soil}$$

with: c0 = $\mu\text{g Ba/ml}$ in blank
 c1 = $\mu\text{g Ba/ml}$ in percolate
 V1 = volume in ml
 V2 =
 10 = dilution constant
 z = valence

11

The soil or clay is first saturated with barium ions from a solution buffered at pH 8.1 with triethanolamine. The barium ions are then exchanged against magnesium ions, and the difference between the initial and final concentrations of magnesium gives a measure of cation exchange capacity. It is usual to remove soluble salts of all kinds before attempting to measure the CEC of the soil. The final measurement may be made by a titration procedure or atomic absorption spectrophotometry.

Procedure:

- A. Common to both methods of determination.
 1. Weigh 5 g $\pm$ 0.001 g of <2mm air-dried soil into a labelled, polymer centrifuge bottle. Note mass in grams of bottle plus soil (M1).
 2. If the soil is NON-CALCAREOUS AND NON-SALINE proceed to (3), otherwise:
 - a) Calcareous soil: Treat soil with 100 ml of buffered bariumchloride reagent for 1 hour to 1 hour 10 min, shaking approx. every 10 min sufficiently to mix the suspension thoroughly. Centrifuge at RCF 415 for 15 min and discard the supernatant liquid, taking care not to lose any soil. If the suspension is turbid, centrifuge further until clear. Proceed as in (3).
 - b) Saline soil: Add 100 ml of 50% (V/V) aqueous ethanol, and shake the suspension thoroughly for 10 min. Centrifuge at 2(a), observing the same precautions. Repeat this procedure until the supernatant liquid is free of soluble salts, e.g. no reaction of acidified silver nitrate solutions to chloride ions. If the soil is calcareous, proceed as in 2(a); if not, proceed as in (3).
- c) IF THE SOIL IS CALCAREOUS AND SALINE THEN BOTH (b) AND (a) MUST BE FOLLOWED IN SEQUENCE WITH (b) FIRST.
 3. Add 200 ml of buffered barium chloride reagent, shake thoroughly and leave the suspension to stand overnight. Centrifuge at RCF 415 or 15 min and discard the supernatant.
 4. Add 200 ml of demineralised water, shake thoroughly and centrifuge as before IMMEDIATELY. Discard the supernatant.
 5. Weigh the bottle and contents (M2).
 6. Pipette 100 ml of 0.25M magnesium sulphate solution into the bottle, and shake the stoppered bottle at approx. 10 min intervals over a 2 hour period. Centrifuge as above, ensuring that the supernatant is completely clear. Stand the bottle on the bench without disturbing the sediment. Determination by titration.
- B. Determination by titration.
 1. Pipette a 5 ml aliquot of the clear supernatant into a labelled, clean, dry 50 ml conical flask, add 5 ml of

19. Base saturation(%)

1 Reference. UNEP - UN/ECE METHOD 9106SA.

Σ base cations * 100 / CEC.
With (K, Ca, Mg and Na) and CEC determined according to the resp. reference methods.

2..14 The calculation method for all laboratories is similar to the reference method (Σ base cations * 100 / CEC), except for labcode 18, they use following equation: $\Sigma(\text{Ca}, \text{Mg}, \text{K}, \text{Na}) * 100 / \Sigma(\text{Ca}, \text{Mg}, \text{K}, \text{Na}, \text{H})$. However the differences between these lab's are caused by there different measuring procedures for the CEC and the exchange (base) cations. Therefore the method description for the base saturation can not be regarded separately from the method descriptions of these 2 parameters. The same list as for the CEC (cfr supra) is applied. 3 remarks can be made:

1. method 6 is not applicable for the base saturation since the corresponding lab doesn't use this CEC value for its calculation (lab 9, they use the reference CEC to calculate the reference BS).
2. method 14 is unspecified, in this case it means that there is no CEC/BS method described or that there is no CEC measured, so it is not clear how the BS is calculated.
3. method 13 is added since labcode 20 reports its national method for Base Saturation but not for the CEC.

$$BS = \frac{\text{sum basic cations}}{\text{sum basic cations} + \text{exch. acidity}}$$

ammonia buffer solution and approx. 0.5 ml of screened indicator.

2. Titrate this solution with the EDTA solution; the end point is orange to apple-green. Call the titre Al.

3. Titrate a 5 ml aliquot of the 0.25M magnesium sulphate solution similarly. The titre for this should be close to 12.5 ml. If it is not, then repeat.

C. Determination by AAS.

1. Pipette 0.2 ml of the centrifuged solution into a 100 ml volumetric flask. Add 5 ml 12M hydrochloric acid, make to volume with demineralised water and mix thoroughly.

2. Set up the AAS according to the instruction handbook.

3. Prepare a calibration graph using the standards prepared above.

4. Measure the concentration of Mg in the unknown solutions.

Calculation:

a) For the titration:

$$\text{corrected titre (A2)} = \text{Al}[(100 + \text{M2-M1})/100] \text{ ml}$$

$$\text{CEC of the air-dry soil} = 8 (\text{B-A2}) \text{ mEq/100 g}$$

If the CEC exceeds 50 mEq/100 g the determination should be repeated using less soil and the calculation adjusted accordingly.

b) For the AAS method:

An accurately prepared solution of 0.025M magnesium sulphate heptahydrate contains 60 mg Mg/100 ml of solution. The actual amount in this solution should be measured and calculated. Call this B.

Al = 50a where a = measured Mg content in the diluted aliquot

Al is corrected for entrained water from the wash step:

$$A2 = \text{Al}(100 + \text{M2-M1})/100 \text{ mg Mg}$$

$$\text{CEC} = [(\text{B-A2})/12] * 20 \text{ mEq/100 g of soil}$$

Report on oven-dry basis.

12 The CEC is calculated by adding the exchangeable cations and the soil unsaturation.

13 unspecified.

20. Total Nitrogen (mg/kg)

1 Reference. UNEP -UN/ECE METHOD 9105SA.
Dry combustion.

2 Kjeldahl.

labcode 10: Principle: The N in the sample is converted to ammonium by wet digestion with concentrated H_2SO_4 in presence of catalyst mixture. NH_4^+ are determined by spectrophotometer TECHNICON AutoAnalyser II.
Procedure: Place 0.4 g of soil sample (for O-horizons 0.2 g) gently ground into a digestion tube. Add about 200 mg catalyst mixture (5 g Se + 20 g $CuSO_4$ + 80 g K_2SO_4) and 5 ml conc. H_2SO_4 , swirl the tube. Place the digestion tube into the digestion heating block (Technicon BD-40) heated to 370°C. After 2.5 hours reaction, take the tube out of the block and allow to cool. Then add carefully a small amount of DI-water, swirl the tube and make to volume 75 ml with DI-water, after cooling. Swirl the tube again and allow to sedimentate overnight. Take supernatant to analyse on Technicon AutoAnalyser II, use the required reagents according to the manual.

labcode 11: The total N amount (mg/kg) is measured by Kjeldahl method with VAPODEST-5 (Gerhard) apparatus.

labcode 15: Kjeldahl-Lauro. The measurement is completely automated, destruction takes place in a Kjeltex destruction block.

Principle: Weigh +/- 4 g of soil sample in destruction tubes. Add 15 ml conc. H_2SO_4 and 1 Ti tablet. Heat the destruction block to 400°C and leave it for 1 hour. Transfer the destruction tubes into an automatic Kjeltex machine. All ammonium is distilled by steam and titrated automatically. Calculation: for 5 g of soil sample:
N-content = # ml HCl 0.1N * 28 mg/100 g dry soil

3 Sample digestion with conc. H_2SO_4 in the presence of a catalyst. Distillation in H_3BO_3 and back-titration with standard H_2SO_4 (Bremner, 1965).

4 Total ICP-AES analysis. The sample is digested in 10 ml of acid mixture (72% $HClO_4$: 70% HNO_3 1:5 V:V) in closed teflon bomb. The bomb is heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety release valve. The results are reported in μmol or $mmol/kg$ dry basis.

5 The sample is wet-ashed in concentrated H_2SO_4 . Oxidation is assisted by addition of 30% H_2O_2 . Any nitrogen present is converted to $(NH_4)_2SO_4$. N is determined colorimetrically.

Weigh 0.5 g of homogenised (<2mm) sample into a 100 ml pyrex volumetric flask. Add 5 ml of concentrated H_2SO_4 using a dispenser. If possible allow to stand overnight to diminish foaming. Same day carry out a moisture factor determination on the sample. Prior to heating, add a few drops of H_2O_2 and when reaction subsides

repeat process. Continue until no more reaction takes place (5 ml H_2O_2). Place on a hotplate and heat at approx. 150°C. Remove from hotplate. Allow to cool and add further H_2O_2 . Return to hotplate and increase temperature to maximum (200°C). Remove after 30 minutes and add further H_2O_2 if required. The sample digest solution should be clear when digesting and when cool. Make to 100 ml volume mark when cool and shake well. A blank and control sample should always be included in every batch, if not several of each depending on batch size.

N is determined colorimetrically using a SKALAR auto analysis flow system.

6 AFNOR X31-111. Determination of total N.

7 Thermal combustion with oxygen. Separation of the combustion gas with separation columns and detection in a heat conductivity cell. Procedure: 40-50 g (for organic samples: 7-10 g) are weighed into tin containers which are closed with a special tweezer. Within 40 samples 8 standards (acetanilide) are measured. The measurement is carried out with an automatic sampler.

8 Determination and detection on a Carlo Erba 1508.

9 unspecified.

transferred from the Teflon vessel to a 50 ml graduated flask over filter paper in glass funnels. The Teflon vessel is rinsed five times with H₂O (deminerallized) which is also transferred to the graduated flask. The filter paper is put into a porcelain crucible and incinerated in a muffle furnace. The weight of the residue is estimated. In the digestion solution the elements are measured directly with ICP and/or AAS (flame or graphite furnace). ICP and/or AAS are calibrated with acidified standard solutions. The digestion solutions of the humus samples are put into an autosampler and measured automatically. P is measured with ICP. K and Ca are measured with ICP or AAS (flame).

Extractant HF

Combustion with H₂SO₄ and HClO₄.

The sample is wet-ashed in concentrated H₂SO₄. Oxidation is assisted by addition of 30% H₂O₂. Any nitrogen present is converted to (NH₄)₂SO₄, P to H₃PO₄, K to K₂SO₄, Ca to CaSO₄ and Mg to MgSO₄. Nitrogen and phosphorus forms are determined colorimetrically and the concentration of the different cations is determined by AAS. Procedure: Weigh 0.5 g of homogenised (<2mm) sample into a 100 ml pyrex volumetric flask. Add 5 ml of concentrated H₂SO₄ using a dispenser. If possible allow to stand overnight to diminish foaming. Same day carry out a moisture factor determination on the sample. Prior to heating add a few drops of H₂O₂ and when reaction subsides repeat process. Continue until no more reaction takes place (5 ml H₂O₂). Place on a hotplate and heat at approx. 150°C. Remove from hotplate. Allow to cool and add further H₂O₂ if required. Return to hotplate and increase temperature to max (200°C). Remove after 30 minutes and add further H₂O₂ if required. The sample digest solution should be clear when digesting and also when cool. Make to 100 ml volume mark when cool and shake well. A blank and control sample should always be included in every batch, if not several of each depending on batch size.

Mg is determined by AAS from the final solution of the modified Mechlich method (determination of exchangeable cations, 0.2N BaCl₂ at pH 8.1).

The dried and ground sample having a particle size of less than 150 µm is ignited at 450°C for 4 hours. After cooling, concentrated fluorhydric and perchloric acids are added to the material and evaporated to dryness. The residue is dissolved in hydrochloric acid and then diluted with water. The element contents of the extract are determined using listed methods: Ca: AAF, K: AEF, P: colorimetric method.

unspecified

21...25. P, K, Ca, Mg and Na (mg/kg)

1 Aqua regia.

2 Extractant: HClO₄ and HNO₃
labcode 2: sample digestion with conc. nitric and perchloric acid.
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 v:v) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety valve. The results are reported in µmol or mmol/kg dry basis.

labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destruction tubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destruction block (+/- 300°C) let the sample react until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid in all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.

2M HNO₃
labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
labcode 18: using AAS-flame.

Dry ashing procedure. Exactly about 2-4 g air dry soil ashed using a LECO TGA-500 analyser. The sample is first heated to 105°C (10°C/min) and maintained until constant weight (moisture content). The temperature is then raised to 550°C (5°C/min) and maintained until constant weight is achieved. The ash is then moistened with 2-3 ml HCl (1 part conc. HCl + 1 part dist. H₂O) and transferred to a boiling waterbath. After the HCl has evaporated off, the sample is dissolved in 10 ml 2N HCl and left to stand, covered with a watch glass, on a boiling water bath for c. 20 min. The sample is then filtered, ashing the residue into the filter paper with 2N HCl. 2N HCl is added to make up 100 ml. The concentrations of above elements were determined using ICP-AES.

Digestion. Principle: The humus sample is digested with HNO₃ under pressure. In the digestion solution the cations and P are measured with ICP and/or AAS.
Procedure: 100-200 mg of the sample is weighed into teflon vessels to which 2 ml HNO₃ is added. After that the vessels are put into the digestion block and heated up slowly to 170°C in a heating cupboard for 10 hours overnight. After cooling down the digestion solution is

automatically. Fe and Zn are measured with ICP or AAS (flame).

7 Extractant HF

8 Extraction with 2N HCl

9 Minéralisation avec des acides concentrés oxydants. On pèse 1 g de sol broyé fin dans un verre "Berzelius" de 100 cm³. On ajoute 10 cm³ de mélange d'acides: 70% HNO₃, 70% HClO₄, 97% H₂SO₄ en rapport de 4:2:0.2. On couvre les solutions avec un verre de montre et on les laisse à la température du chambre jusqu'au deuxième jour. Puis, on met les verres "Berzelius" sur un bain de sable, un peu découvert, on les laisse jusqu'au sec et encore quelque temps pour évaporation totale de SO₃. On répète ces opérations avec 5 cm³ de mélange d'acides. On les laisse sur le bain de sable encore 2-3 heures pour l'insolubilisation de la silice. Après la réfrigération, on ajoute de HCl, 2 ml pour les échantillons de sol et 1 ml pour les échantillons végétaux, et 2-3 cm³ de l'eau distillée bouillante et on laisse au chaud 5-10 min. On filtre par papier filtrant dans un ballon de 50 cm³. On rince le verre et le filtre avec de l'eau distillée, jusqu'au moment quand le Cl⁻ passe en solution. On remplit le ballon jusqu'au signe avec de l'eau distillée. On dose au spectrophotomètre en absorption atomique: Cu, Zn, Pb, Co, Ni, Mn, Cd, Cr, Fe, Mg.

10 unspecified

30..32. Cu (mg/kg) and Pb (mg/kg) and Mn (mg/kg)

1 Aqua regia

2 Extractant: HClO₄ and HNO₃
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 V:V) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety valve. The results are reported in µmol or mmol/kg dry basis.

labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destruction tubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destruction block (+/- 300°C) let the sample react, until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid in all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to

volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.

2M HNO₃
labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
labcode 18: using AAS-flame.

4 AFNOR X31-151. Solubilising of metallic trace elements by acid attacks. Method n° 2: fluorhydric acid.

5 Dry ashing procedure. Exactly about 2-4 g air dry soil ashed using a LECO TGA-500 analyser. The sample is first heated to 105°C (10°C/min) and maintained until constant weight (moisture content). The temperature is then raised to 550°C (5°C/min) and maintained until constant weight is achieved. The ash is then moistened with 2-3 ml HCl (1 part conc. HCl + 1 part dist. H₂O) and transferred to a boiling waterbath. After the HCl has evaporated off, the sample is dissolved in 10 ml 2N HCl and left to stand, covered with a watch glass, on a boiling water bath for c. 20 min. The sample is then filtered, ashing the residue into the filter paper with 2N HCl. 2N HCl is added to make up 100 ml. The concentrations of above elements are determined using ICP-AES.

6 Digestion. Principle: The humus sample is digested with HNO₃ under pressure. In the digestion solution the cations are measured with ICP and/or AAS. Procedure: 100-200 mg of the sample is weighed into teflon vessels to which 2 ml HNO₃ is added. After that the vessels are put into the digestion block and heated up slowly to 170°C in a heating cupboard for 10 hours overnight.

After cooling down, the digestion solution is transferred from the Teflon vessel to a 50 ml graduated flask over filter paper in glass funnels. The Teflon vessel is rinsed five times with H₂O (deminerallized) which is also transferred to the graduated flask. The filter paper is put into a porcelain crucible and incinerated in a muffle furnace. The weight of the residue is estimated. In the digestion solution the elements are measured directly with ICP and/or AAS (flame or graphite furnace). ICP and/or AAS are calibrated with acidified standard solutions. The digestion solutions of the humus samples are put into an autosampler and measured automatically. Mn and Cu are measured with ICP or AAS (flame). Sometimes AAS with graphite furnace is needed to measure Cu.

Extraction with 2N HCl

Minéralisation avec des acides concentrés oxydants. On pèse 1 g de sol broyé fin dans un verre "Berzelius" de 100 cm³. On ajoute 10 cm³ de mélange d'acides: 70% HNO₃, 70% HClO₄, 97% H₂SO₄ en rapport de 4:2:0.2. On couvre avec un verre de montre et on laisse à la température du chambre jusqu'au deuxième jour.

26. Sulfur S (mg/kg)

1 S-LECO

2 Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 V:V) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety valve. The results are reported in μmol or mmol/kg dry basis.

3 Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.

4 Total content. X-ray fluorescence.

5 Dry combustion.

6 Totale destructie met HClO₄, 2*2.5 g staal en 2*1 g staal, gemeten met calorimeter bij 420 nm in 1 cm cuvetten. Omrekening sulfaat naar zwavel.

7 Digestion. Principle: The humus sample is digested with HNO₃ under pressure. In the digestion solution the S is measured with ICP. Procedure: 100-200 mg of the sample is weighed into teflon vessels to which 2 ml HNO₃ is added. After that the vessels are put into the digestion block and heated up slowly to 170°C in a heating cupboard for 10 hours overnight. After cooling down the digestion solution is transferred from the Teflon vessel to a 50 ml graduated flask over filter paper in glass funnels. The Teflon vessel is rinsed five times with H₂O (deminerallized) which is also transferred to the graduated flask. The filter paper is put into a porcelain crucible and incinerated in a muffle furnace. The weight of the residue is estimated. In the digestion solution the S is measured directly with ICP. ICP is calibrated with acidified standard solutions. The digestion solutions of the humus samples are put into an autosampler and measured automatically.

27..29. Al, Fe and Zn (mg/kg)

1 Aqua regia.

2 Extractant: HClO₄ and HNO₃
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 V:V) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety valve. The results are reported in μmol or mmol/kg dry basis.

labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destructiontubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destructionblock (+/- 300°C) let the sample react until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid in all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.

2M HNO₃

labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
labcode 18: using AAS-flame.

4 AFNOR X31-151. Solubilising of metallic trace elements by acid attacks. Method n° 2: fluorhydric acid.

5 Dry ashing procedure. Exactly about 2-4 g air dry soil ashed using a LECO TGA-500 analyser. The sample is first heated to 105°C (10°C/min) and maintained until constant weight (moisture content). The temperature is then raised to 550°C (5°C/min) and maintained until constant weight is achieved. The ash is then moistened with 2-3 ml HCl (1 part conc. HCl + 1 part dist. H₂O) and transferred to a boiling waterbath. After the HCl has evaporated off, the sample is dissolved in 10 ml 2N HCl and left to stand, covered with a watch glass, on a boiling water bath for c. 20 min. The sample is then filtered, ashing the residue into the filter paper with 2N HCl. 2N HCl is added to make up 100 ml. The concentrations of above elements were determined using ICP-AES.

6 Digestion. Principle: The humus sample is digested with HNO₃ under pressure. In the digestion solution the cations are measured with ICP and/or AAS. Procedure: 100-200 mg of the sample is weighed into teflon vessels to which 2 ml HNO₃ is added. After that the vessels are put into the digestion block and heated up slowly to 170°C in a heating cupboard for 10 hours overnight. After cooling down the digestion solution is transferred from the Teflon vessel to a 50 ml graduated flask over filter paper in glass funnels. The Teflon vessel is rinsed five times with H₂O (deminerallized) which is also transferred to the graduated flask. The filter paper is put into a porcelain crucible and incinerated in a muffle furnace. The weight of the residue is estimated. In the digestion solution the elements are measured directly with ICP and/or AAS (flame or graphite furnace). ICP and/or AAS are calibrated with acidified standard solutions. The digestion solutions of the humus samples are put into an autosampler and measured

d'acides: 70% HNO₃, 70% HClO₄, 97% H₂SO₄ en rapport de 4:2:0.2. On couvre avec un verre de montre et on laisse à la température du chambre jusqu'au deuxième jour. Puis, on met les verres "Berzelius" sur un bain de sable, un peu découvert, on les laisse jusqu'au sec et répète ces opérations avec 5 cm³ de mélange d'acides. On les laisse sur le bain de sable encore 2-3 heures pour l'insolubilisation de la silice. Après la réfrigération, on ajoute de HCl, 2 ml pour les échantillons de sol et 1 ml pour les échantillons végétaux, et 2-3 cm³ de l'eau distillée bouillante et on laisse au chaud 5-10 min. On filtre par papier filtrant dans un ballon de 50 cm³. On rince le verre et le filtre avec de l'eau distillée, jusqu'au moment quand le Cl⁻ passe en solution. On remplit le ballon jusqu'au signe avec de l'eau distillée. On dose au spectrophotomètre en absorption atomique: Cu, Zn, Pb, Co, Ni, Mn, Cd, Cr, Fe, Mg.

unspecified

6

Puis, on met les verres "Berzelius" sur un bain de sable, un peu découvert, on les laisse jusqu'au sec et encore quelque temps pour évaporation totale de SO₃. On répète ces opérations avec 5 cm³ de mélange d'acides. On les laisse sur le bain de sable encore 2-3 heures pour l'insolubilisation de la silice. Après la réfrigération, on ajoute de HCl, 2 ml pour les échantillons de sol et 1 ml pour les échantillons végétaux, et 2-3 cm³ de l'eau distillée bouillante et on laisse au chaud 5-10 min. On filtre par papier filtrant dans un ballon de 50 cm³. On rince le verre et le filtre avec de l'eau distillée, jusqu'au moment quand le Cl⁻ passe en solution. On remplit le ballon jusqu'au signe avec de l'eau distillée. On dose au spectrophotomètre en absorption atomique: Cu, Zn, Pb, Co, Ni, Mn, Cd, Cr, Fe, Mg.

unspecified

33. Cr (mg/kg)

1 Aqua regia.

2 Extractant: HClO₄ and HNO₃
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 V:V) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gas escape through the safety valve. The results are reported in $\mu\text{mol/kg}$ dry basis.

labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destruction tubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destruction block (+/- 300°C) let the sample react until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid an all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.

3 2M HNO₃
labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
labcode 18: using AAS-flame.

4 AFNOR X31-151. Solubilising of metallic trace elements by acid attacks. Method n° 2: fluorhydric acid.

5 Minéralisation avec des acides concentrés oxydants. On pèse 1 g de sol broyé fin dans un verre "Berzelius" de 100 cm³. On ajoute 10 cm³ de mélange

34. Ni (mg/kg)

- 1 Aqua regia.
- 2 Extractant: HClO₄ and HNO₃
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 v:v) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gases escape through the safety valve. The results are reported in μmol or mmol/kg dry basis.
labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destruction tubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destruction block (+/- 300°C) let the sample react until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid an all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.

3 2M HNO₃

- labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
- labcode 18: using AAS-flame.

4 AFNOR X31-151. Solubilising of metallic trace elements by acid attacks. Method n° 2: fluorhydric acid.

- 5 Minéralisation avec des acides concentrés oxydants. On pèse 1 g de sol broyé fin dans un verre "Berzelius" de 100 cm³. On ajoute 10 cm³ de mélange d'acides: 70% HNO₃, 70% HClO₄, 97% H₂SO₄ en rapport de 4:2:0.2. On couvre avec un verre de montre et on laisse à la température du chambre jusqu'au deuxième jour. Puis, on met les verres "Berzelius" sur un bain de sable, un peu découvert, on les laisse jusqu'au sec et encore quelque temps pour évaporation totale de SO₃. On répète ces opérations avec 5 cm³ de mélange d'acides. On les laisse sur le bain de sable encore 2-3 heures pour l'insolubilisation de la silice. Après la réfrigération, on ajoute de HCl, 2 ml pour les échantillons de sol et 1 ml pour les échantillons végétaux, et 2-3 cm³ de l'eau distillée bouillante et on laisse au chaud 5-10 min. On filtre par papier filtrant dans un ballon de 50 cm³. On rince le verre et le filtre avec de l'eau distillée, jusqu'au moment quand le Cl⁻ passe en solution. On remplit le ballon jusqu'au signe avec de l'eau distillée. On dose au spectrophotomètre en absorption atomique: Cu, Zn, Pb, Co, Ni, Mn, Cd, Cr, Fe, Mg.

35. Cd (mg/kg)

- 1 Aqua regia.
- 2 Extractant: HClO₄ and HNO₃
labcode 3: Total analysis ICP-AES. The sample is digested in 10 ml of acid mixture (70% HNO₃, 72% HClO₄, 1:5 v:v) in closed teflon bomb. The bomb is then heated in a microwave oven. The temperature is controlled so only negligible amounts of gases escape through the safety valve. The results are reported in μmol or mmol/kg dry basis.
labcode 9: acid digestion, 5 parts HNO₃ and 1 part HClO₄
labcode 15: total destruction. Weigh 2*2.5 g sample and 2*1 g sample into destruction tubes. Add 15 ml conc. H₂SO₄ and 2.5 ml HClO₄. Prepare some blanks too. Heat the destruction block (+/- 300°C) let the sample react until nearly dry. If not all the material has reacted, cool the samples off and add an extra amount of conc. nitric acid an all the tubes. Heat again (300°C) until reaction takes place. Remove all the tubes from the destruction block and add a small amount of 1N HNO₃. Filter the samples warm in 100 ml flasks and make to volume mark with 1N HNO₃. The cations are determined by AAS or ICP-AES.
- 3 2M HNO₃
labcode 4: Total analysis. 2M HNO₃ * 5 g in 50 ml * 2 hours in boiling waterbath.
labcode 18: using AAS-flame.
- 4 AFNOR X31-151. Solubilising of metallic trace elements by acid attacks. Method n° 2: fluorhydric acid.
- 5 Dry ashing procedure. Exactly about 2-4 g air dry soil ashed using a LECO TGA-500 analyser. The sample is first heated to 105°C (10°C/min) and maintained until constant weight (moisture content). The temperature is then raised to 550°C (5°C/min) and maintained until constant weight is achieved. The ash is then moistened with 2-3 ml HCl (1 part conc. HCl + 1 part dist. H₂O) and transferred to a boiling waterbath. After the HCl has evaporated off, the sample is dissolved in 10 ml 2N HCl and left to stand, covered with a watch glass, on a boiling water bath for c. 20 min. The sample is then filtered, ashing the residue into the filter paper with 2N HCl. 2N HCl is added to make up 100 ml. The concentrations of above elements were determined using ICP-AES.
- 6 Digestion. Principle: The humus sample is digested with HNO₃ under pressure. In the digestion solution the cations with ICP or AAS. Procedure: 100-200 mg of the sample is weighed into teflon vessels to which 2 ml HNO₃ is added. After that the vessels are put into the digestion block and heated up slowly to 170°C in a

heating cupboard for 10 hours overnight. After cooling down the digestion solution is transferred from the Teflon vessel to a 50 ml graduated flask over filter paper in glass funnels. The Teflon vessel is rinsed five times with H_2O (demineralized) which is also transferred to the graduated flask. The filter paper is put into a porcelain crucible and incinerated in a muffle furnace. The weight of the residue is estimated. In the digestion solution the elements are measured directly with ICP or AAS (flame or graphite furnace). ICP or AAS are calibrated with acidified standard solutions. The digestion solutions of the humus samples are put into an autosampler and measured automatically. For Cd AAS with graphite furnace is needed.

Minéralisation avec des acides concentrés oxydants. On pèse 1 g de sol broyé fin dans un verre "Berzelius" de 100 cm³. On ajoute 10 cm³ de mélange d'acides: 70% HNO_3 , 70% $HClO_4$, 97% H_2SO_4 en rapport de 4:2:0.2. On couvre avec un verre de montre et on laisse à la température du chambre jusqu'au deuxième jour. Puis, on met les verres "Berzelius" sur un bain de sable, on peu découvrir, on les laisse jusqu'au sec et encore quelque temps pour évaporation totale de SO_3 . On répète ces opérations avec 5 cm³ de mélange d'acides. On les laisse sur un bain de sable encore 2-3 heures pour l'insolubilisation de la silice. Après la réfrigération, on ajoute de HCl , 2 ml pour les échantillons de sol et 1 ml pour les échantillons végétaux, et 2-3 cm³ de l'eau distillée bouillante et on laisse au chaud 5-10 min. On filtre par papier filtrant dans un ballon de 50 cm³. On rince le verre et le filtre avec de l'eau distillée, jusqu'au moment quand le Cl^- passe en solution. On remplit le ballon jusqu'au signe avec de l'eau distillée. On dose au spectrophotomètre en absorption atomique: Cu, Zn, Pb, Co, Ni, Mn, Cd, Cr, Fe, Mg.

36. Hg (mg/kg)

- 1 Aqua regia, AAS-continuous flow vapour system (Hydrid).
- 2 2M HNO_3 , AAS-continuous flow vapour system (Hydrid).
- 3 AFNOR X31-151. Solubilizing of metallic trace elements by acid attacks - method n°2: fluorhydric acid (major & minor elements are analysed on the same extract).

Part II : INTERCALIBRATION

This intercalibration study is set up to investigate the effect of the different methods on the analysis results of a set of soil parameters.

Data for 36 parameters are processed separately, but in all cases the strategy is the same. First, the input data of each laboratory are described and summarised (TABLE I, II and III). Next, regardless of the laboratory, the results are grouped according to the method. For each method group, mean values are calculated (TABLE IV + graphics). Multiple comparisons of the mean group values are performed using ANOVA and TUKEY's honest significantly difference (TABLE V).

2.1. Results of data processing

TABLE I : Overview of the reported data

This table informs about the laboratories ("labcode") that have measured a certain parameter. The number of replicates ("repl") and the used soil samples ("samples reported") are also shown. The laboratories that did not report a number of replicates receive the record "1 not reported". Their reported values are mean values, the result of at least 1 measurement. If there is a reference method prescribed, then an additional column is added for that specific parameter namely "N/R method". Some laboratories only measure according to their national method (N) or to the reference method (R), others use both (N/R). It also happens that a laboratory's national method is the reference method (N=R), in which case its (N) results are classified in the reference group.

TABLE II : Summary method descriptions

The different methods are shortly described by means of a few key-words and they receive a code-number. This table only serves as a quick-reference for interpreting the results in following tables. For further detail, see the complete list of method descriptions in Part I.

TABLE III (a, b) : Descriptive statistics

Some descriptive statistics are calculated for each laboratory and they are grouped according to the sample (A1, A2, B1, B2). In case there is a reference method, a separate table (IIIB) is added. The "method" column (only included in the national tables) refers to the method-number, several laboratories can use a similar national method so they have the same number. The number of replicates is shown in the "repl" column. The minimum, mean and maximum values

each other or not, it compares the variance between the methods with the variance within the method-groups. A large F-value gives little support to the statement that the different methods are comparable. If $\text{prob} > F$ is less or equal than 0.05, you can say with 95% confidence that there are significant differences between a number of methods. However, a significant F-test does not provide information on which methods differ from which other methods. To point these out, a lot of multiple comparison procedures are available. Here the Tukey's honest significantly difference test (Snedecar & Cochran, 1967) is performed to compare all pairs of means. The results are also shown in TABLE V. Confidence intervals are very useful in multiple comparisons because they show the degree of uncertainty in each comparison in an easy way. The null hypothesis states that the true difference between 2 means equals zero. By constructing a confidence interval that does indeed bracket 0, you have 95% chance that this interval is correct and that the calculated difference between the compared means can indeed equal zero. Remember that a failure to reject the null hypothesis implies that (i) the means are indeed different or (ii) the difference between the method means are not large enough to be detected with the given sample size ("repl" in table IV).

The 36 parameters are worked out in order according to table I in PART I.

are simple statistics that can be easily derived from the reported data. The standard deviation ("std") is a measure of variation within a laboratory. Small values correspond with slight differences between the replicates. The coefficient of variation ("cv") measures the relative variability. std and cv are generally low, this reflects the precision with which the measurements are performed by the participating laboratories. Remark the blank fields when only 1 replicate or a mean value is reported.

The accompanying graphs show the minimum, mean and maximum values for each laboratory, sample and method. The laboratories ("labcode") are placed in the x-axis, the measured parameter values in the y-axis. There are separated graphics for the reference and the national methods. In the national method graphics the method-numbers are drawn at the top of the bars. This clarifies the visual inspection of the differences between the laboratories.

TABLE IV : Total mean and normal distribution

To verify which methods differ from one another or from the reference method, all laboratories with the same method number are first placed together. Their results are treated as 1 random sample. Mean values and standard deviations ("std") are calculated for each group of methods. The Shapiro-Wilks statistic tests the normality of the distribution of these compiled method samples. The closer this value ("normal") to 1, the more support is given to the hypothesis that the input data values are indeed a random sample from a normal population. The amount of support is quantitatively expressed by the $\text{prob} < W$ - value. Multiplied by 100, this figure expresses the probability (in percentage) to correctly describe the sample as a normal distribution.

TABLE V : Method comparison

TABLE IV addresses the question whether the results obtained by different methods for a given parameter are on average different from the reference method or from the other involved methods.

The above question is answered by conducting the general linear models (GLM) procedure. This unbalanced ANOVA tests how well the methods, considered as the only source of variation, can explain the differences in the reported parameter values. The 4 samples (A1, A2, B1 and B2) are treated as subgroups and are processed separately. GLM computes some interesting statistics among which 2 are very important, namely the F-test and R^2 . R^2 measures how much variation in the dependent variable (parameter) can be accounted for by the model (parameter = method). A high R^2 indicates that the methods do have an important influence on the measured parameter values, R^2 is low when other sources of variation are more important. The F-test for equal means indicates whether the means of the different methods are significantly different from

1. pH (CaCl2)

units: -
mandatory for all samples
reported by 22 laboratories

TABLE I : pH (CaCl2) - Overview of the reported data

labcode	samples reported	number of replicates	N/R method
1	A1 A2 B1 B2	3	N/R
2	A1 A2 B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	R A2: N
5	A1 A2 B1	2	R
6	A1 A2 B1 B2	2	N/R
7	A1 A2 B1 B2	3	N/R
8	A1 A2 B1 B2	3	R
9	A1 A2 B1 B2	3	R
10	A1 A2 B1 B2	3	R
11	A1 A2 B1 B2	3	R
12	A1 A2 B1 B2	3	N
13	A1 A2 B1 B2	3	N=R
14	A1 A2 B1 B2	3	R
15	A1 A2 B1 B2	2	N/R
16	A1 A2 B1 B2	3	N
17	A1 A2 B1 B2	3	R
18	A1 A2 B1 B2	3	R
19	A1 A2 B1 B2	3	R
20	A1 A2 B1 B2	1 not reported	R
21	A1 A2 B1 B2	3	N/R
22	A1 A2 B1 B2	3	N/R

TABLE II : pH (CaCl2) - Summary method descriptions

method	description
1	reference method 1:5, for H- & O- horizons 1:20
2	CaCl2 1:5, for H- & O-horizons 1:1
3	AFNOR X 31-103
4	CaCl2 1:2.5
5	CaCl2 1:25, for H- & O-horizons 1:5
6	CaCl2 0.01M
7	CaCl2 0.01M 1:2
8	KCl 0.1M 1:5
9	KCl 1N
10	KCl 1N 1:2.5
11	unspecified

TABLE IIIa : pH (CaCl2) - Descriptive statistics

sample A1 - national methods

labocde	method	repl	minimum	mean	maximum	std	cv
1	9	3	7.21	7.21	7.21	0.00	0.00
2	2	3	7.02	7.09	7.13	0.06	0.90
3	6	5	7.60	7.62	7.63		
4							
5							
6							
7	3	3	7.64	7.65	7.65	0.01	0.08
8	11	3	7.00	7.00	7.00	0.00	0.00
9							
10							
11							
12							
13	11	3	7.51	7.54	7.58	0.04	0.47
14							
15							
16	10	3	7.29	7.32	7.34	0.03	0.38
17	5	3	6.62	6.63	6.63	0.01	0.09
18							
19							
20							
21	4	3	7.50	7.58	7.63	0.07	0.92
22	8	3	7.43	7.44	7.46	0.02	0.23

sample A2 - national methods

labocde	method	repl	minimum	mean	maximum	std	cv
1	9	3	4.51	4.52	4.53	0.01	0.26
2	2	3	4.64	4.69	4.75	0.06	1.17
3	6	5	4.60	4.61	4.62		
4	7	3	4.55	4.56	4.57	0.01	0.22
5							
6							
7	3	3	4.57	4.58	4.60	0.02	0.38
8	11	3	3.90	3.90	3.90	0.00	0.00
9							
10							
11							
12							
13	11	3	4.71	4.71	4.71	0.00	0.00
14							
15							
16	10	3	4.55	4.57	4.61	0.03	0.70
17	5	3	4.41	4.42	4.44	0.02	0.39
18							
19							
20							
21	4	3	4.60	4.62	4.63	0.02	0.38
22	8	3	4.37	4.38	4.39	0.01	0.23

sample B1 - national methods

labocde	method	repl	minimum	mean	maximum	std	cv
1	9	3	3.50	3.60	3.68	0.09	2.49
2	2	3	3.56	3.56	3.58	0.00	0.00
3	6	5	3.80	3.80	3.81		
4							
5							
6							
7	3	3	3.81	3.81	3.82	0.01	0.15
8	11	3	2.90	2.95	3.00	0.05	1.70
9							
10							
11							
12							
13	11	3	3.91	3.91	3.92	0.01	0.15
14							
15							
16	10	3	3.56	3.57	3.58	0.01	0.32
17	5	3	3.77	3.77	3.77	0.00	0.00
18							
19							
20							
21	4	3	3.83	3.84	3.84	0.01	0.15
22	8	3	3.56	3.56	3.57	0.01	0.18

sample B2 - national methods

labocde	method	repl	minimum	mean	maximum	std	cv
1	9	3	3.44	3.48	3.52	0.04	1.16
2	2	3	3.35	3.40	3.43	0.05	1.36
3	6	5	3.55	3.55	3.56		
4							
5							
6							
7	3	3	3.71	3.73	3.75	0.02	0.54
8	11	3	2.70	2.77	2.80	0.06	2.09
9							
10							
11							
12							
13	11	3	3.67	3.67	3.68	0.01	0.16
14							
15							
16	10	3	3.42	3.44	3.46	0.02	0.61
17	5	3	3.50	3.51	3.51	0.01	0.17
18							
19							
20							
21	4	3	3.60	3.61	3.62	0.01	0.28
22	8	3	3.41	3.43	3.44	0.02	0.45

TABLE IIIb : pH (CaCl2) - Descriptive statistics

sample A1 - reference method

labco	repl	minimum	mean	maximum	std	cv
1	3	7.28	7.31	7.35	0.04	0.49
2						
3						
4	4	7.50	7.51	7.51	0.01	0.07
5	1		7.43			
6	4	6.89	7.24	7.61	0.39	5.43
7	3	7.51	7.59	7.66	0.08	1.01
8	3	7.00	7.00	7.00	0.00	0.00
9	3	7.52	7.54	7.55	0.02	0.20
10	3	7.57	7.58	7.59	0.01	0.13
11	3	7.07	7.08	7.10	0.02	0.22
12	3	7.23	7.26	7.27	0.02	0.32
13						
14	3	7.28	7.29	7.31	0.02	0.21
15	2	7.10	7.10	7.10	0.00	0.00
16	3	7.41	7.42	7.43	0.01	0.16
17						
18	3	7.61	7.63	7.65	0.02	0.27
19	3		7.33			
20	1		7.40			
21	3	7.65	7.69	7.72	0.04	0.47
22	3	7.49	7.49	7.50	0.01	0.08

sample A2 - reference method

labco	repl	minimum	mean	maximum	std	cv
1	3	4.42	4.46	4.50	0.04	0.91
2						
3						
4						
5	1		4.63			
6	4	4.59	4.60	4.60	0.01	0.13
7	3	4.57	4.61	4.65	0.04	0.88
8	3	3.90	3.90	3.91	0.01	0.15
9	3	4.55	4.58	4.63	0.04	0.91
10	3	4.57	4.59	4.60	0.02	0.33
11	3	4.54	4.58	4.63	0.05	1.00
12	3	4.59	4.60	4.61	0.01	0.22
13						
14	3	4.56	4.57	4.58	0.01	0.22
15	2	4.60	4.60	4.60	0.00	0.00
16	3	4.59	4.61	4.64	0.03	0.63
17						
18	3	4.87	4.94	4.97	0.06	1.17
19	3		4.64			
20	1		4.60			
21	3	3.47	3.50	3.55	0.04	1.19
22	3	4.52	4.52	4.53	0.01	0.13

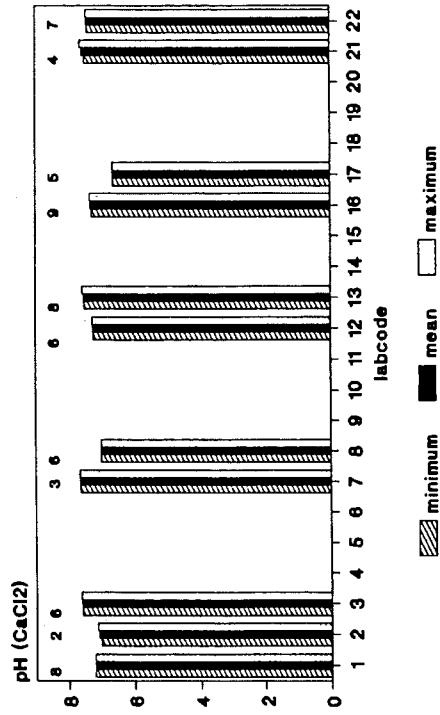
sample B1 - reference method

labco	repl	minimum	mean	maximum	std	cv
1	3	3.74	3.76	3.77	0.02	0.41
2						
3						
4	4	3.75	3.76	3.77	0.01	0.22
5	1		3.69			
6	4	3.89	3.90	3.91	0.01	0.21
7	3	3.87	3.88	3.88	0.01	0.15
8	3	2.90	2.95	3.00	0.05	1.70
9	3	3.77	3.79	3.82	0.03	0.66
10	3	3.80	3.82	3.84	0.02	0.54
11	3	3.89	3.70	3.70	0.01	0.16
12	3	3.87	3.88	3.89	0.01	0.26
13						
14	3	3.77	3.78	3.78	0.01	0.15
15	2	3.90	3.90	3.90	0.00	0.00
16	3	3.87	3.88	3.89	0.01	0.26
17						
18	3	3.94	3.97	4.03	0.05	1.31
19	3		3.97			
20	1		4.00			
21	3	3.67	3.68	3.69	0.01	0.31
22	3	3.72	3.74	3.76	0.02	0.56

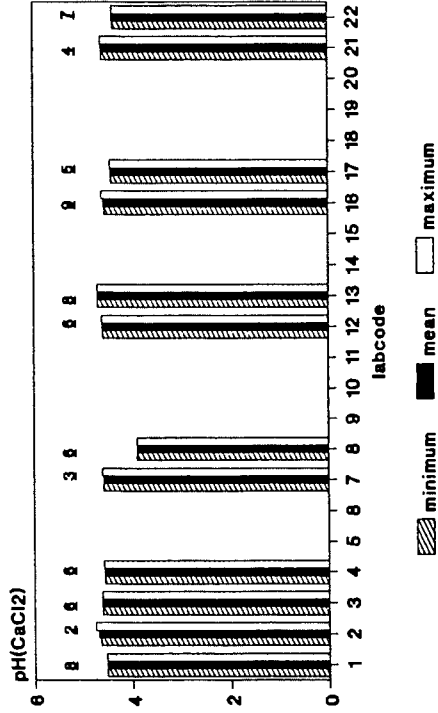
sample B2 - reference method

labco	repl	minimum	mean	maximum	std	cv
1	3	3.56	3.56	3.57	0.01	0.16
2						
3						
4	4	3.52	3.55	3.56	0.02	0.49
5						
6	4	3.59	3.61	3.61	0.01	0.28
7	3	3.60	3.64	3.73	0.08	2.06
8	3	2.70	2.77	2.80	0.06	2.09
9	3	3.57	3.59	3.61	0.02	0.56
10	3	3.64	3.65	3.67	0.02	0.42
11	3	3.45	3.48	3.51	0.03	0.86
12	3	3.59	3.61	3.62	0.02	0.42
13						
14	3	3.50	3.50	3.51	0.01	0.17
15	2	3.60	3.60	3.60	0.00	0.00
16	3	3.61	3.62	3.63	0.01	0.32
17						
18	3	3.61	3.63	3.66	0.03	0.73
19	3		3.62			
20	1		3.75			
21	3	3.45	3.47	3.50	0.03	0.83
22	3	3.48	3.49	3.50	0.01	0.33

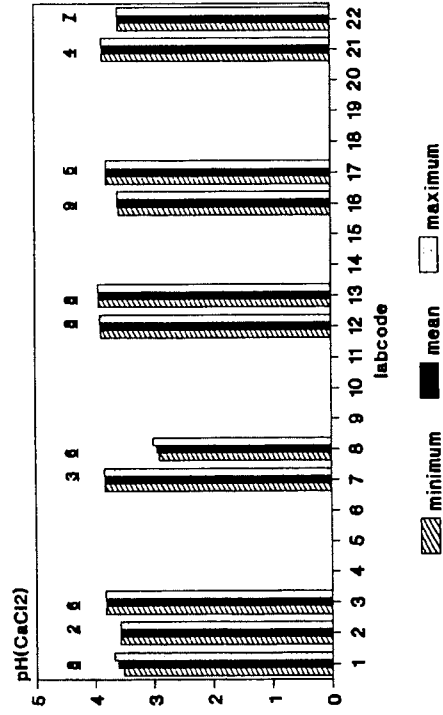
pH sample A1
national methods



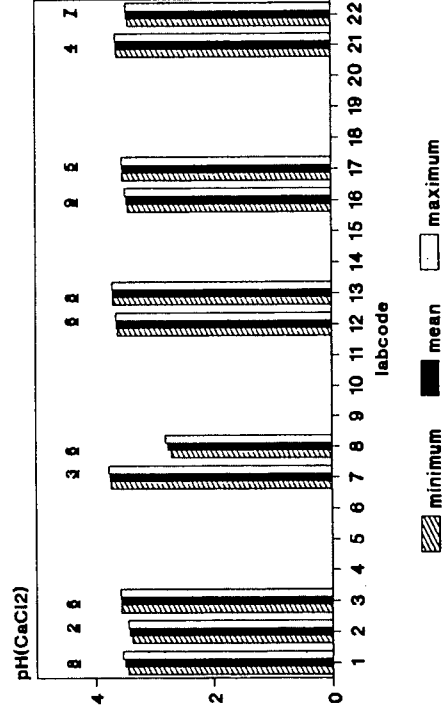
pH sample A2
national methods



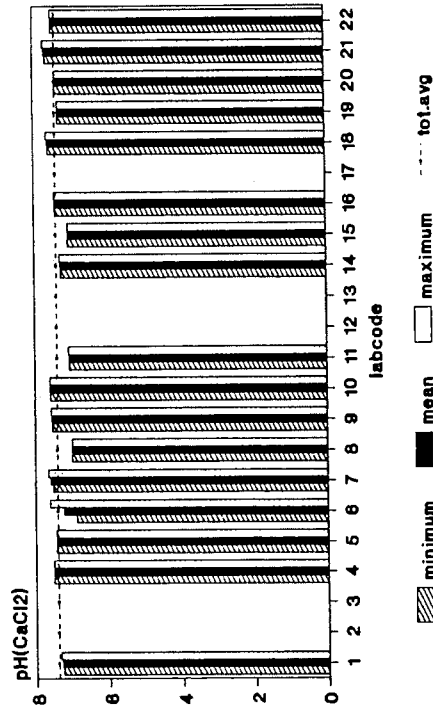
pH sample B1
national methods



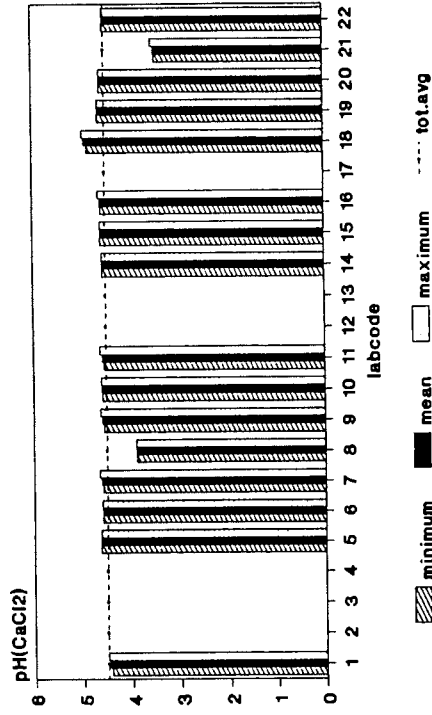
pH sample B2
national methods



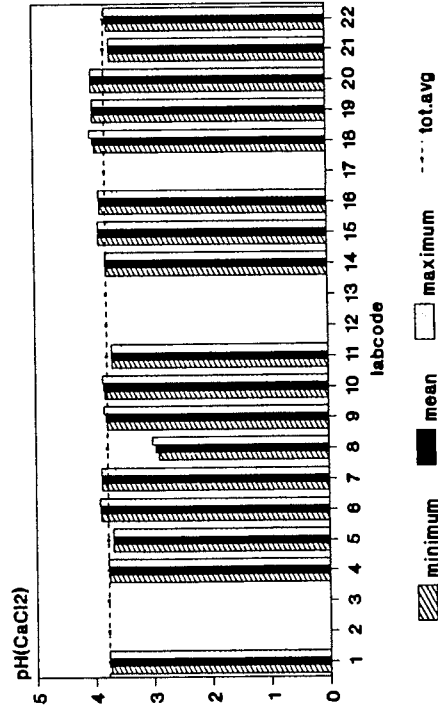
pH sample A1
reference method



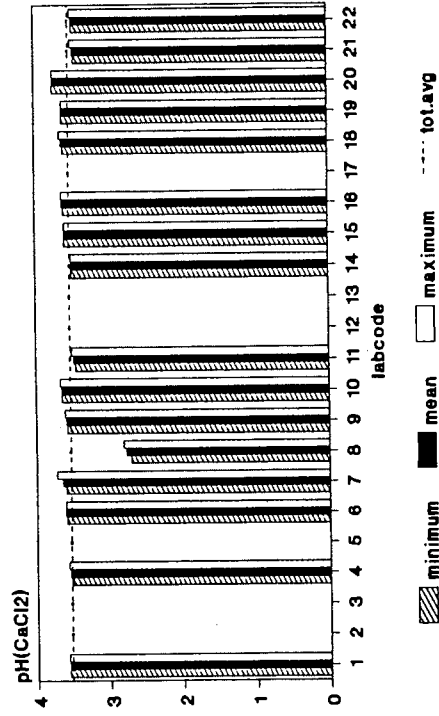
pH sample A2
reference method



pH sample B1
reference method



pH sample B2
reference method



----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 4 5 6 8 9

Number of observations in by group = 74

Dependent Variable: pH(CaCl2)

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	2.42812101	6.07	0.0001
Error	64	2.84272899		
Corrected Total	73	5.27085000		
R-Square		C.V.		pH(CaCl2)
Mean		0.460670	2.869366	7.34500000

Source	DF	Type I SS	F Value	Pr > F
METH	9	2.42812101	6.07	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	2.42812101	6.07	0.0001

TABLE IV : pH (CaCl2) - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	46	7.38	0.23	0.92	0.01
A1	2	3	7.09	0.06	0.75	0.00
A1	3	3	7.65	0.01	0.75	0.00
A1	4	3	7.58	0.07	0.86	0.27
A1	5	3	6.63	0.01	0.75	0.00
A1	6	5	7.62			
A1	8	3	7.44	0.02	0.75	0.00
A1	9	3	7.21	0.00	0.00	0.00
A1	10	3	7.32	0.03	0.89	0.36
A1	11	6	7.27	0.30	0.73	0.01
A2	1	42	4.48	0.35	0.67	0.00
A2	2	3	4.69	0.06	1.00	0.90
A2	3	3	4.58	0.02	0.75	0.00
A2	4	3	4.62	0.02	0.75	0.00
A2	5	3	4.42	0.02	0.75	0.00
A2	6	5	4.61			
A2	7	3	4.56	0.01	1.00	0.99
A2	8	3	4.38	0.01	1.00	0.99
A2	9	3	4.52	0.01	0.75	0.00
A2	10	3	4.57	0.03	0.87	0.30
A2	11	6	4.31	0.44	0.68	0.00
A3	1	3	4.54	0.01	0.75	0.00
B1	1	46	3.77	0.24	0.64	0.00
B1	2	3	3.56	0.00	0.00	0.00
B1	3	3	3.81	0.01	0.75	0.00
B1	4	3	3.84	0.01	0.75	0.00
B1	5	3	3.77	0.00	0.00	0.00
B1	6	5	3.80			
B1	8	3	3.56	0.01	0.75	0.00
B1	9	3	3.60	0.09	0.80	0.11
B1	10	3	3.57	0.01	0.75	0.00
B1	11	6	3.43	0.53	0.72	0.01
B2	1	45	3.53	0.22	0.57	0.00
B2	2	3	3.40	0.05	0.75	0.00
B2	3	3	3.73	0.02	1.00	0.99
B2	4	3	3.61	0.01	1.00	0.99
B2	5	3	3.51	0.01	0.75	0.00
B2	6	6	3.55			
B2	8	3	3.43	0.02	0.96	0.64
B2	9	3	3.48	0.04	0.98	0.73
B2	10	3	3.44	0.02	0.92	0.46
B2	11	6	3.22	0.50	0.73	0.01

Remark:
Labcode 3 and labcode 19 reported mean values for resp. 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for methods 6 and 1 are different from the real number of replicates.

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: pH(CaCl2)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 0.044418
Critical Value of Studentized Range= 4.635

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
3 - 6	-0.7710	0.0267	0.8243	-0.7710	0.0267	0.8243
3 - 4	-0.4974	0.0667	0.6307	-0.4974	0.0667	0.6307
3 - 8	-0.3574	0.2067	0.7707	-0.3574	0.2067	0.7707
3 - 1	-0.1461	0.2656	0.6772	-0.1461	0.2656	0.6772
3 - 10	-0.2374	0.3267	0.8907	-0.2374	0.3267	0.8907
3 - 11	-0.1135	0.3750	0.8635	-0.1135	0.3750	0.8635
3 - 9	-0.1274	0.4367	1.0007	-0.1274	0.4367	1.0007
3 - 2	-0.0107	0.5533	1.1174	-0.0107	0.5533	1.1174
3 - 5	0.4560	1.0200	1.5840	0.4560	1.0200	1.5840
6 - 3	-0.8243	-0.0267	0.7710	-0.8243	-0.0267	0.7710
6 - 4	-0.7577	0.0400	0.8377	-0.7577	0.0400	0.8377
6 - 8	-0.6177	0.1800	0.9777	-0.6177	0.1800	0.9777
6 - 1	-0.4593	0.2389	0.9372	-0.4593	0.2389	0.9372
6 - 10	-0.4977	0.3000	1.0977	-0.4977	0.3000	1.0977
6 - 11	-0.3978	0.3483	1.0945	-0.3978	0.3483	1.0945
6 - 9	-0.3877	0.4100	1.2077	-0.3877	0.4100	1.2077
6 - 2	-0.2710	0.5267	1.3243	-0.2710	0.5267	1.3243
6 - 5	0.1957	0.9933	1.7910	0.1957	0.9933	1.7910
4 - 3	-0.6307	-0.0667	0.4974	-0.6307	-0.0667	0.4974
4 - 6	-0.8377	-0.0400	0.7577	-0.8377	-0.0400	0.7577
4 - 8	-0.4240	0.1400	0.7040	-0.4240	0.1400	0.7040
4 - 1	-0.2127	0.1989	0.6105	-0.2127	0.1989	0.6105
4 - 10	-0.3040	0.2600	0.8240	-0.3040	0.2600	0.8240
4 - 11	-0.1801	0.3083	0.7968	-0.1801	0.3083	0.7968
4 - 9	-0.1940	0.3700	0.9340	-0.1940	0.3700	0.9340
4 - 2	-0.0774	0.4867	1.0507	-0.0774	0.4867	1.0507
4 - 5	0.3893	0.9533	1.5174	0.3893	0.9533	1.5174
8 - 3	-0.7707	-0.2067	0.3574	-0.7707	-0.2067	0.3574
8 - 6	-0.9777	-0.1800	0.6177	-0.9777	-0.1800	0.6177
8 - 4	-0.7040	-0.1400	0.4240	-0.7040	-0.1400	0.4240
8 - 1	-0.3527	0.0589	0.4705	-0.3527	0.0589	0.4705
8 - 10	-0.4440	0.1200	0.6840	-0.4440	0.1200	0.6840
8 - 11	-0.3201	0.1683	0.6568	-0.3201	0.1683	0.6568
8 - 9	-0.3340	0.2300	0.7940	-0.3340	0.2300	0.7940
8 - 2	-0.2174	0.3467	0.9107	-0.2174	0.3467	0.9107
8 - 5	0.2493	0.8133	1.3774	0.2493	0.8133	1.3774

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 3	-0.6772	-0.2656	0.1461	-0.6772	-0.2656	0.1461
1 - 6	-0.9372	-0.2389	0.4593	-0.9372	-0.2389	0.4593
1 - 4	-0.6105	-0.1989	0.2127	-0.6105	-0.1989	0.2127
1 - 8	-0.4705	-0.0589	0.3527	-0.4705	-0.0589	0.3527
1 - 10	-0.3505	0.0611	0.4727	-0.3505	0.0611	0.4727
1 - 11	-0.1904	0.1094	0.4093	-0.1904	0.1094	0.4093
1 - 9	-0.2405	0.1711	0.5827	-0.2405	0.1711	0.5827
1 - 2	-0.1239	0.2878	0.6994	-0.1239	0.2878	0.6994
1 - 5	0.3428	0.7544	1.1661	0.3428	0.7544	1.1661
10 - 3	-0.8907	-0.3267	0.2374	-0.8907	-0.3267	0.2374
10 - 6	-1.0977	-0.3000	0.4977	-1.0977	-0.3000	0.4977
10 - 4	-0.8240	-0.2600	0.3040	-0.8240	-0.2600	0.3040
10 - 8	-0.6840	-0.1200	0.4440	-0.6840	-0.1200	0.4440
10 - 1	-0.4727	-0.0611	0.3505	-0.4727	-0.0611	0.3505
10 - 11	-0.4401	0.0483	0.5368	-0.4401	0.0483	0.5368
10 - 9	-0.4540	0.1100	0.6740	-0.4540	0.1100	0.6740
10 - 2	-0.3374	0.2267	0.7907	-0.3374	0.2267	0.7907
10 - 5	0.1293	0.6933	1.2574	0.1293	0.6933	1.2574
11 - 3	-0.8635	-0.3750	0.1135	-0.8635	-0.3750	0.1135
11 - 6	-1.0945	-0.3483	0.3978	-1.0945	-0.3483	0.3978
11 - 4	-0.7968	-0.3083	0.1801	-0.7968	-0.3083	0.1801
11 - 8	-0.6568	-0.1683	0.3201	-0.6568	-0.1683	0.3201
11 - 1	-0.4093	-0.1094	0.1904	-0.4093	-0.1094	0.1904
11 - 10	-0.5368	-0.0483	0.4401	-0.5368	-0.0483	0.4401
11 - 9	-0.4268	0.0617	0.5501	-0.4268	0.0617	0.5501
11 - 2	-0.3101	0.1783	0.6668	-0.3101	0.1783	0.6668
11 - 5	0.1565	0.6450	1.1335	0.1565	0.6450	1.1335
9 - 3	-1.0007	-0.4367	0.1274	-1.0007	-0.4367	0.1274
9 - 6	-1.2077	-0.4100	0.3877	-1.2077	-0.4100	0.3877
9 - 4	-0.9340	-0.3700	0.1940	-0.9340	-0.3700	0.1940
9 - 8	-0.7940	-0.2300	0.3340	-0.7940	-0.2300	0.3340
9 - 1	-0.5827	-0.1711	0.2405	-0.5827	-0.1711	0.2405
9 - 10	-0.6740	-0.1100	0.4540	-0.6740	-0.1100	0.4540
9 - 11	-0.5501	-0.0617	0.4268	-0.5501	-0.0617	0.4268
9 - 2	-0.4474	0.1167	0.6807	-0.4474	0.1167	0.6807
9 - 5	0.0193	0.5833	1.1474	0.0193	0.5833	1.1474
2 - 3	-1.1174	-0.5533	0.0107	-1.1174	-0.5533	0.0107
2 - 6	-1.3243	-0.5267	0.2710	-1.3243	-0.5267	0.2710
2 - 4	-1.0507	-0.4867	0.0774	-1.0507	-0.4867	0.0774
2 - 8	-0.9107	-0.3467	0.2174	-0.9107	-0.3467	0.2174
2 - 1	-0.6994	-0.2878	0.1239	-0.6994	-0.2878	0.1239
2 - 10	-0.7907	-0.2267	0.3374	-0.7907	-0.2267	0.3374
2 - 11	-0.6668	-0.1783	0.3101	-0.6668	-0.1783	0.3101
2 - 9	-0.6807	-0.1167	0.4474	-0.6807	-0.1167	0.4474

pH (CaCl2) Method comparison

pH (CaCl2) Method comparison

SAMPLE=A1

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
2 - 5	-0.0974	0.4667	1.0307	
5 - 3	-1.5840	-1.0200	-0.4560	***
5 - 6	-1.7910	-0.9933	-0.1957	***
5 - 4	-1.5174	-0.9533	-0.3893	***
5 - 8	-1.3774	-0.8133	-0.2493	***
5 - 1	-1.1661	-0.7544	-0.3428	***
5 - 10	-1.2574	-0.6933	-0.1293	***
5 - 11	-1.1335	-0.6450	-0.1565	***
5 - 9	-1.1474	-0.5833	-0.0193	***
5 - 2	-1.0307	-0.4667	0.0974	

Number of observations in by group = 74

NOTE: Due to missing values, only 73 observations can be used in this analysis.

Dependent Variable: pH(CaCl2)

Source	DF	Sum of Squares	F Value	Pr > F
Model	10	0.50972577	0.53	0.8594
Error	62	5.91736190		
Corrected Total	72	6.42708767		
R-Square				
C.V.				
pH(CaCl2)				
Mean				
		6.881160		4.48958904

Source	DF	Type I SS	F Value	Pr > F
METH	10	0.50972577	0.53	0.8594
Source	DF	Type III SS	F Value	Pr > F
METH	10	0.50972577	0.53	0.8594

----- SAMPLE=A2 -----

----- SAMPLE=A2 -----

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: pH(CaCl2)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 62 MSE= 0.095441
Critical Value of Studentized Range= 4.726

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
2 - 4	-0.7696	0.0733	0.9162	-0.5196	0.0974	0.7143
2 - 6	-1.1087	0.0833	1.2754	-0.6829	0.1600	1.0029
2 - 3	-0.7296	0.1133	0.9562	-0.6429	0.2000	1.0429
2 - 10	-0.7229	0.1200	0.9629	-0.4550	0.2750	1.0050
2 - 7	-0.7096	0.1333	0.9762			
2 - 9	-0.6662	0.1767	1.0196			
2 - 1	-0.4062	0.2107	0.8277	-0.9629	-0.1200	0.7229
2 - 5	-0.5696	0.2733	1.1162	-0.8896	-0.0467	0.7962
2 - 8	-0.5296	0.3133	1.1562	-1.2287	-0.0367	1.1554
2 - 11	-0.3416	0.3883	1.1183	-0.8496	-0.0067	0.8362
4 - 2	-0.9162	-0.0733	0.7696	-0.8296	0.0133	0.8562
4 - 6	-1.1821	0.0100	1.2021	-0.7862	0.0567	0.8996
4 - 3	-0.8029	0.0400	0.8829	-0.5262	0.0907	0.7077
4 - 10	-0.7962	0.0467	0.8896	-0.6896	0.1533	0.9962
4 - 7	-0.7829	0.0600	0.9029	-0.6496	0.1933	1.0362
4 - 9	-0.7396	0.1033	0.9462	-0.4616	0.2683	0.9983
4 - 1	-0.4796	0.1374	0.7543	-0.9762	-0.1333	0.7096
4 - 5	-0.6429	0.2000	1.0429	-0.9029	-0.0600	0.7829
4 - 8	-0.6029	0.2400	1.0829	-1.2421	-0.0500	1.1421
4 - 11	-0.4150	0.3150	1.0450	-0.8629	-0.0200	0.8229
6 - 2	-1.2754	-0.0833	1.1087	-0.8562	-0.0133	0.8296
6 - 4	-1.2021	-0.0100	1.1821	-0.7996	0.0433	0.8862
6 - 3	-1.1621	0.0300	1.2221	-0.5396	0.0774	0.6943
6 - 10	-1.1554	0.0367	1.2287	-0.7029	0.1400	0.9829
6 - 7	-1.1421	0.0500	1.2421	-0.6629	0.1800	1.0229
6 - 9	-1.0987	0.0933	1.2854	-0.4750	0.2550	0.9850
6 - 1	-0.9172	0.1274	1.1719	-1.0196	-0.1767	0.6662
6 - 5	-1.0021	0.1900	1.3821	-0.9462	-0.1033	0.7396
6 - 8	-0.9621	0.2300	1.4221	-1.2854	-0.0933	1.0987
6 - 11	-0.8101	0.3050	1.4201	-0.9062	-0.0633	0.7796
3 - 2	-0.9562	-0.1133	0.7296	-0.8996	-0.0567	0.7862
3 - 4	-0.8829	-0.0400	0.8029	-0.8862	-0.0433	0.7996
3 - 6	-1.2221	-0.0300	1.1621	-0.5829	0.0340	0.6510
3 - 10	-0.8362	0.0067	0.8496	-0.7462	0.0967	0.9396
3 - 7	-0.8229	0.0200	0.8629	-0.7062	0.1367	0.9796
3 - 9	-0.7796	0.0633	0.9062	-0.5183	0.2117	0.9416
3 - 1						
3 - 5				-0.8277	-0.2107	0.4062
3 - 8				-0.7543	-0.1374	0.4796
3 - 11				-1.1719	-0.1274	0.9172
				-0.7143	-0.0974	0.5196
				-0.7077	-0.0907	0.5262
				-0.6943	-0.0774	0.5396
				-0.6510	-0.0340	0.5829
				-0.5543	0.0626	0.6796
				-0.5143	0.1026	0.7196
				-0.2729	0.1776	0.6282

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
3 - 1	-0.5196	0.0974	0.7143			
3 - 5	-0.6829	0.1600	1.0029			
3 - 8	-0.6429	0.2000	1.0429			
3 - 11	-0.4550	0.2750	1.0050			
10 - 2	-0.9629	-0.1200	0.7229			
10 - 4	-0.8896	-0.0467	0.7962			
10 - 6	-1.2287	-0.0367	1.1554			
10 - 3	-0.8496	-0.0067	0.8362			
10 - 7	-0.8296	0.0133	0.8562			
10 - 9	-0.7862	0.0567	0.8996			
10 - 1	-0.5262	0.0907	0.7077			
10 - 5	-0.6896	0.1533	0.9962			
10 - 8	-0.6496	0.1933	1.0362			
10 - 11	-0.4616	0.2683	0.9983			
7 - 2	-0.9762	-0.1333	0.7096			
7 - 4	-0.9029	-0.0600	0.7829			
7 - 6	-1.2421	-0.0500	1.1421			
7 - 3	-0.8629	-0.0200	0.8229			
7 - 10	-0.8562	-0.0133	0.8296			
7 - 9	-0.7996	0.0433	0.8862			
7 - 1	-0.5396	0.0774	0.6943			
7 - 5	-0.7029	0.1400	0.9829			
7 - 8	-0.6629	0.1800	1.0229			
7 - 11	-0.4750	0.2550	0.9850			
9 - 2	-1.0196	-0.1767	0.6662			
9 - 4	-0.9462	-0.1033	0.7396			
9 - 6	-1.2854	-0.0933	1.0987			
9 - 3	-0.9062	-0.0633	0.7796			
9 - 10	-0.8996	-0.0567	0.7862			
9 - 7	-0.8862	-0.0433	0.7996			
9 - 1	-0.5829	0.0340	0.6510			
9 - 5	-0.7462	0.0967	0.9396			
9 - 8	-0.7062	0.1367	0.9796			
9 - 11	-0.5183	0.2117	0.9416			
1 - 2	-0.8277	-0.2107	0.4062			
1 - 4	-0.7543	-0.1374	0.4796			
1 - 6	-1.1719	-0.1274	0.9172			
1 - 3	-0.7143	-0.0974	0.5196			
1 - 10	-0.7077	-0.0907	0.5262			
1 - 7	-0.6943	-0.0774	0.5396			
1 - 9	-0.6510	-0.0340	0.5829			
1 - 5	-0.5543	0.0626	0.6796			
1 - 8	-0.5143	0.1026	0.7196			
1 - 11	-0.2729	0.1776	0.6282			

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 4 5 6 8 9

Number of observations in by group = 74

Dependent Variable: pH(CaCl2)

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	0.94163850	1.71	0.1054
Error	64	3.92021014		
Corrected Total	73	4.86184865		
Mean				
		R-Square	C.V.	pH(CaCl2)
		0.193679	6.662261	3.71486486
Source	DF	Type I SS	F Value	Pr > F
METH	9	0.94163850	1.71	0.1054
Source	DF	Type III SS	F Value	Pr > F
METH	9	0.94163850	1.71	0.1054

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Limit	Confidence Limit	Difference Between Means	Upper Limit
5 - 2	-1.1162	-0.2733	0.5696	0.5696
5 - 4	-1.0429	-0.2000	0.6429	0.6429
5 - 6	-1.3821	-0.1900	1.0021	1.0021
5 - 3	-1.0029	-0.1600	0.6829	0.6829
5 - 10	-0.9962	-0.1533	0.6896	0.6896
5 - 7	-0.9829	-0.1400	0.7029	0.7029
5 - 9	-0.9396	-0.0967	0.7462	0.7462
5 - 1	-0.6796	-0.0626	0.5543	0.5543
5 - 8	-0.8029	0.0400	0.8829	0.8829
5 - 11	-0.6150	0.1150	0.8450	0.8450
8 - 2	-1.1562	-0.3133	0.5296	0.5296
8 - 4	-1.0829	-0.2400	0.6029	0.6029
8 - 6	-1.4221	-0.2300	0.9621	0.9621
8 - 3	-1.0429	-0.2000	0.6429	0.6429
8 - 10	-1.0362	-0.1933	0.6496	0.6496
8 - 7	-1.0229	-0.1800	0.6629	0.6629
8 - 9	-0.9796	-0.1367	0.7062	0.7062
8 - 1	-0.7196	-0.1026	0.5143	0.5143
8 - 8	-0.8829	-0.0400	0.8029	0.8029
8 - 11	-0.6550	0.0750	0.8050	0.8050
11 - 2	-1.1183	-0.3883	0.3416	0.3416
11 - 4	-1.0450	-0.3150	0.4150	0.4150
11 - 6	-1.4201	-0.3050	0.8101	0.8101
11 - 3	-1.0050	-0.2750	0.4550	0.4550
11 - 10	-0.9983	-0.2683	0.4616	0.4616
11 - 7	-0.9850	-0.2550	0.4750	0.4750
11 - 9	-0.9416	-0.2117	0.5183	0.5183
11 - 1	-0.6282	-0.1776	0.2729	0.2729
11 - 8	-0.8450	-0.1150	0.6150	0.6150
11 - 11	-0.8050	-0.0750	0.6550	0.6550

----- SAMPLE=B1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: pH(CaCl2)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 0.061253
Critical Value of Studentized Range= 4.635

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 3	-0.6390	0.0233	0.6857	-0.6390	0.0233	0.6857
4 - 6	-0.9000	0.0367	0.9734	-0.9000	0.0367	0.9734
4 - 5	-0.5957	0.0667	0.7290	-0.5957	0.0667	0.7290
4 - 1	-0.4152	0.0682	0.5516	-0.4152	0.0682	0.5516
4 - 9	-0.4290	0.2333	0.8957	-0.4290	0.2333	0.8957
4 - 10	-0.3990	0.2633	0.9257	-0.3990	0.2633	0.9257
4 - 8	-0.3890	0.2733	0.9357	-0.3890	0.2733	0.9357
4 - 2	-0.3857	0.2767	0.9390	-0.3857	0.2767	0.9390
4 - 11	-0.1686	0.4050	0.9786	-0.1686	0.4050	0.9786
3 - 4	-0.6857	-0.0233	0.6390	-0.6857	-0.0233	0.6390
3 - 6	-0.9234	0.0133	0.9500	-0.9234	0.0133	0.9500
3 - 5	-0.6190	0.0433	0.7057	-0.6190	0.0433	0.7057
3 - 1	-0.4385	0.0449	0.5282	-0.4385	0.0449	0.5282
3 - 9	-0.4524	0.2100	0.8724	-0.4524	0.2100	0.8724
3 - 10	-0.4224	0.2400	0.9024	-0.4224	0.2400	0.9024
3 - 8	-0.4124	0.2500	0.9124	-0.4124	0.2500	0.9124
3 - 2	-0.4090	0.2533	0.9157	-0.4090	0.2533	0.9157
3 - 11	-0.1919	0.3817	0.9553	-0.1919	0.3817	0.9553
6 - 4	-0.9734	-0.0367	0.9000	-0.9734	-0.0367	0.9000
6 - 3	-0.9500	-0.0133	0.9234	-0.9500	-0.0133	0.9234
6 - 5	-0.9067	0.0300	0.9667	-0.9067	0.0300	0.9667
6 - 1	-0.7885	0.0315	0.8515	-0.7885	0.0315	0.8515
6 - 9	-0.7400	0.1967	1.1334	-0.7400	0.1967	1.1334
6 - 10	-0.7100	0.2267	1.1634	-0.7100	0.2267	1.1634
6 - 8	-0.7000	0.2367	1.1734	-0.7000	0.2367	1.1734
6 - 2	-0.6967	0.2400	1.1767	-0.6967	0.2400	1.1767
6 - 11	-0.5079	0.3683	1.2445	-0.5079	0.3683	1.2445
5 - 4	-0.7290	-0.0667	0.5957	-0.7290	-0.0667	0.5957
5 - 3	-0.7057	-0.0433	0.6190	-0.7057	-0.0433	0.6190
5 - 6	-0.9667	-0.0300	0.9067	-0.9667	-0.0300	0.9067
5 - 1	-0.4819	0.0015	0.4849	-0.4819	0.0015	0.4849
5 - 9	-0.4957	0.1667	0.8290	-0.4957	0.1667	0.8290
5 - 10	-0.4657	0.1967	0.8590	-0.4657	0.1967	0.8590
5 - 8	-0.4557	0.2067	0.8690	-0.4557	0.2067	0.8690
5 - 2	-0.4524	0.2100	0.8724	-0.4524	0.2100	0.8724
5 - 11	-0.2353	0.3383	0.9119	-0.2353	0.3383	0.9119

----- SAMPLE=B1 -----

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 4	-0.5516	-0.0682	0.4152	-0.5516	-0.0682	0.4152
1 - 3	-0.5282	-0.0449	0.4385	-0.5282	-0.0449	0.4385
1 - 6	-0.8515	-0.0315	0.7885	-0.8515	-0.0315	0.7885
1 - 5	-0.4849	-0.0015	0.4819	-0.4849	-0.0015	0.4819
1 - 9	-0.3182	0.1651	0.6485	-0.3182	0.1651	0.6485
1 - 10	-0.2882	0.1951	0.6785	-0.2882	0.1951	0.6785
1 - 8	-0.2782	0.2051	0.6885	-0.2782	0.2051	0.6885
1 - 2	-0.2749	0.2085	0.6919	-0.2749	0.2085	0.6919
1 - 11	-0.0153	0.3368	0.6889	-0.0153	0.3368	0.6889
9 - 4	-0.8957	-0.2333	0.4290	-0.8957	-0.2333	0.4290
9 - 3	-0.8724	-0.2100	0.4524	-0.8724	-0.2100	0.4524
9 - 6	-1.1334	-0.1967	0.7400	-1.1334	-0.1967	0.7400
9 - 5	-0.8290	-0.1667	0.4957	-0.8290	-0.1667	0.4957
9 - 1	-0.6485	-0.1651	0.3182	-0.6485	-0.1651	0.3182
9 - 10	-0.6324	0.0300	0.6924	-0.6324	0.0300	0.6924
9 - 8	-0.6224	0.0400	0.7024	-0.6224	0.0400	0.7024
9 - 2	-0.6190	0.0433	0.7057	-0.6190	0.0433	0.7057
9 - 11	-0.4019	0.1717	0.7453	-0.4019	0.1717	0.7453
10 - 4	-0.9257	-0.2633	0.3990	-0.9257	-0.2633	0.3990
10 - 3	-0.9024	-0.2400	0.4224	-0.9024	-0.2400	0.4224
10 - 6	-1.1634	-0.2267	0.7100	-1.1634	-0.2267	0.7100
10 - 5	-0.8590	-0.1967	0.4657	-0.8590	-0.1967	0.4657
10 - 1	-0.6785	-0.1951	0.2882	-0.6785	-0.1951	0.2882
10 - 9	-0.6924	-0.0300	0.6324	-0.6924	-0.0300	0.6324
10 - 8	-0.6524	0.0100	0.6724	-0.6524	0.0100	0.6724
10 - 2	-0.6490	0.0133	0.6757	-0.6490	0.0133	0.6757
10 - 11	-0.4319	0.1417	0.7153	-0.4319	0.1417	0.7153
8 - 4	-0.9357	-0.2733	0.3890	-0.9357	-0.2733	0.3890
8 - 3	-0.9124	-0.2500	0.4124	-0.9124	-0.2500	0.4124
8 - 6	-1.1734	-0.2367	0.7000	-1.1734	-0.2367	0.7000
8 - 5	-0.8690	-0.2067	0.4557	-0.8690	-0.2067	0.4557
8 - 1	-0.6885	-0.2051	0.2782	-0.6885	-0.2051	0.2782
8 - 9	-0.7024	-0.0400	0.6224	-0.7024	-0.0400	0.6224
8 - 10	-0.6724	-0.0100	0.6524	-0.6724	-0.0100	0.6524
8 - 2	-0.6590	0.0033	0.6657	-0.6590	0.0033	0.6657
8 - 11	-0.4419	0.1317	0.7053	-0.4419	0.1317	0.7053
2 - 4	-0.9390	-0.2767	0.3857	-0.9390	-0.2767	0.3857
2 - 3	-0.9157	-0.2533	0.4090	-0.9157	-0.2533	0.4090
2 - 6	-1.1767	-0.2400	0.6967	-1.1767	-0.2400	0.6967
2 - 5	-0.8724	-0.2100	0.4524	-0.8724	-0.2100	0.4524
2 - 1	-0.6919	-0.2085	0.2749	-0.6919	-0.2085	0.2749
2 - 9	-0.7057	-0.0433	0.6190	-0.7057	-0.0433	0.6190
2 - 10	-0.6757	-0.0133	0.6490	-0.6757	-0.0133	0.6490
2 - 8	-0.6657	-0.0033	0.6590	-0.6657	-0.0033	0.6590

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 4 5 6 8 9

Number of observations in by group = 74

NOTE: Due to missing values, only 73 observations can be used in this analysis.

Dependent Variable: pH(CaCl2)

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	0.75967793	1.61	0.1323
Error	63	3.3071111		
Corrected Total	72	4.06678904		

R-Square	C.V.	pH(CaCl2)
0.186800	6.548459	3.49876712

Source	DF	Type I SS	F Value	Pr > F
METH	9	0.75967793	1.61	0.1323
Source	DF	Type III SS	F Value	Pr > F
METH	9	0.75967793	1.61	0.1323

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
2 - 11	-0.4453	0.1283	0.7019	
11 - 4	-0.9786	-0.4050	0.1686	
11 - 3	-0.9553	-0.3817	0.1919	
11 - 6	-1.2445	-0.3683	0.5079	
11 - 5	-0.9119	-0.3383	0.2353	
11 - 1	-0.6889	-0.3368	0.0153	
11 - 9	-0.7453	-0.1717	0.4019	
11 - 10	-0.7153	-0.1417	0.4319	
11 - 8	-0.7053	-0.1317	0.4419	
11 - 2	-0.7019	-0.1283	0.4453	

----- SAMPLE=B2 -----

----- SAMPLE=B2 -----

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: pH(CaCl2)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 63 MSE= 0.052494
Critical Value of Studentized Range= 4.638

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
3 - 4	-0.4935	0.1200	0.7335	-0.8602	-0.2467	0.3668
3 - 6	-0.6876	0.1800	1.0476	-0.7402	-0.1267	0.4868
3 - 1	-0.2458	0.2022	0.6503	-0.9343	-0.0667	0.8010
3 - 5	-0.3902	0.2233	0.8368	-0.4925	-0.0444	0.4036
3 - 9	-0.3668	0.2467	0.8602	-0.6368	-0.0233	0.5902
3 - 10	-0.3202	0.2933	0.9068	-0.5668	0.0467	0.6602
3 - 8	-0.3102	0.3033	0.9168	-0.5568	0.0567	0.6702
3 - 2	-0.2868	0.3267	0.9402	-0.5335	0.0800	0.6935
3 - 11	-0.0213	0.5100	1.0413	-0.2680	0.2633	0.7947
4 - 3	-0.7335	-0.1200	0.4935	-0.9068	-0.2933	0.3202
4 - 6	-0.8076	0.0600	0.9276	-0.7868	-0.1733	0.4402
4 - 1	-0.3658	0.0822	0.5303	-0.9810	-0.1133	0.7543
4 - 5	-0.5102	0.1033	0.7168	-0.5392	-0.0911	0.3569
4 - 9	-0.4868	0.1267	0.7402	-0.6835	-0.0700	0.5435
4 - 10	-0.4402	0.1733	0.7868	-0.6602	-0.0467	0.5668
4 - 8	-0.4302	0.1833	0.7968	-0.6035	0.0100	0.6235
4 - 2	-0.4068	0.2067	0.8202	-0.5802	0.0333	0.6468
4 - 11	-0.1413	0.3900	0.9213	-0.3147	0.2167	0.7480
6 - 3	-1.0476	-0.1800	0.6876	-0.9168	-0.3033	0.3102
6 - 4	-0.9276	-0.0600	0.8076	-0.7968	-0.1833	0.4302
6 - 1	-0.7375	0.0222	0.7819	-0.9910	-0.1233	0.7443
6 - 5	-0.8243	0.0433	0.9110	-0.5492	-0.1011	0.3469
6 - 9	-0.8010	0.0667	0.9343	-0.6935	-0.0800	0.5335
6 - 10	-0.7543	0.1133	0.9810	-0.6702	-0.0567	0.5568
6 - 8	-0.7443	0.1233	0.9910	-0.6235	-0.0100	0.6035
6 - 2	-0.7210	0.1467	1.0143	-0.5902	0.0233	0.6368
6 - 11	-0.4816	0.3300	1.1416	-0.3247	0.2067	0.7380
1 - 3	-0.6503	-0.2022	0.2458	-0.9402	-0.3267	0.2868
1 - 4	-0.5303	-0.0822	0.3658	-0.8202	-0.2067	0.4068
1 - 6	-0.7819	-0.0222	0.7375	-1.0143	-0.1467	0.7210
1 - 5	-0.4269	0.0211	0.4692	-0.5725	-0.1244	0.3236
1 - 9	-0.4036	0.0444	0.4925	-0.7168	-0.1033	0.5102
1 - 10	-0.3569	0.0911	0.5392	-0.6935	-0.0800	0.5335
1 - 8	-0.3469	0.1011	0.5492	-0.6468	-0.0333	0.5802
1 - 2	-0.3236	0.1244	0.5725	-0.6368	-0.0233	0.5902
1 - 11	-0.0188	0.3078	0.6343			

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
5 - 3	-0.8368	-0.2233	0.3902	-0.8368	-0.2233	0.3902
5 - 4	-0.7168	-0.1033	0.5102	-0.7168	-0.1033	0.5102
5 - 6	-0.9110	-0.0433	0.8243	-0.9110	-0.0433	0.8243
5 - 1	-0.4692	-0.0211	0.4269	-0.4692	-0.0211	0.4269
5 - 9	-0.5902	0.0233	0.6368	-0.5902	0.0233	0.6368
5 - 10	-0.5435	0.0700	0.6835	-0.5435	0.0700	0.6835
5 - 8	-0.5335	0.0800	0.6935	-0.5335	0.0800	0.6935
5 - 2	-0.5102	0.1033	0.7168	-0.5102	0.1033	0.7168
5 - 11	-0.2447	0.2867	0.8180	-0.2447	0.2867	0.8180
9 - 3	-0.8602	-0.2467	0.3668	-0.8602	-0.2467	0.3668
9 - 4	-0.7402	-0.1267	0.4868	-0.7402	-0.1267	0.4868
9 - 6	-0.9343	-0.0667	0.8010	-0.9343	-0.0667	0.8010
9 - 1	-0.4925	-0.0444	0.4036	-0.4925	-0.0444	0.4036
9 - 5	-0.6368	-0.0233	0.5902	-0.6368	-0.0233	0.5902
9 - 10	-0.5668	0.0467	0.6602	-0.5668	0.0467	0.6602
9 - 8	-0.5568	0.0567	0.6702	-0.5568	0.0567	0.6702
9 - 2	-0.5335	0.0800	0.6935	-0.5335	0.0800	0.6935
9 - 11	-0.2680	0.2633	0.7947	-0.2680	0.2633	0.7947
10 - 3	-0.9068	-0.2933	0.3202	-0.9068	-0.2933	0.3202
10 - 4	-0.7868	-0.1733	0.4402	-0.7868	-0.1733	0.4402
10 - 6	-0.9810	-0.1133	0.7543	-0.9810	-0.1133	0.7543
10 - 1	-0.5392	-0.0911	0.3569	-0.5392	-0.0911	0.3569
10 - 5	-0.6835	-0.0700	0.5435	-0.6835	-0.0700	0.5435
10 - 9	-0.6602	-0.0467	0.5668	-0.6602	-0.0467	0.5668
10 - 8	-0.6035	0.0100	0.6235	-0.6035	0.0100	0.6235
10 - 2	-0.5802	0.0333	0.6468	-0.5802	0.0333	0.6468
10 - 11	-0.3147	0.2167	0.7480	-0.3147	0.2167	0.7480
8 - 3	-0.9168	-0.3033	0.3102	-0.9168	-0.3033	0.3102
8 - 4	-0.7968	-0.1833	0.4302	-0.7968	-0.1833	0.4302
8 - 6	-0.9910	-0.1233	0.7443	-0.9910	-0.1233	0.7443
8 - 1	-0.5492	-0.1011	0.3469	-0.5492	-0.1011	0.3469
8 - 5	-0.6935	-0.0800	0.5335	-0.6935	-0.0800	0.5335
8 - 9	-0.6702	-0.0567	0.5568	-0.6702	-0.0567	0.5568
8 - 10	-0.6235	-0.0100	0.6035	-0.6235	-0.0100	0.6035
8 - 2	-0.5902	0.0233	0.6368	-0.5902	0.0233	0.6368
8 - 11	-0.3247	0.2067	0.7380	-0.3247	0.2067	0.7380
2 - 3	-0.9402	-0.3267	0.2868	-0.9402	-0.3267	0.2868
2 - 4	-0.8202	-0.2067	0.4068	-0.8202	-0.2067	0.4068
2 - 6	-1.0143	-0.1467	0.7210	-1.0143	-0.1467	0.7210
2 - 1	-0.5725	-0.1244	0.3236	-0.5725	-0.1244	0.3236
2 - 5	-0.7168	-0.1033	0.5102	-0.7168	-0.1033	0.5102
2 - 9	-0.6935	-0.0800	0.5335	-0.6935	-0.0800	0.5335
2 - 10	-0.6468	-0.0333	0.5802	-0.6468	-0.0333	0.5802
2 - 8	-0.6368	-0.0233	0.5902	-0.6368	-0.0233	0.5902

units: -
optional for all samples
reported by 16 laboratories

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
2 - 11	-0.3480	0.1833	0.7147	
11 - 3	-1.0413	-0.5100	0.0213	
11 - 4	-0.9213	-0.3900	0.1413	
11 - 6	-1.1416	-0.3300	0.4816	
11 - 1	-0.6343	-0.3078	0.0188	
11 - 5	-0.8180	-0.2867	0.2447	
11 - 9	-0.7947	-0.2633	0.2680	
11 - 10	-0.7480	-0.2167	0.3147	
11 - 8	-0.7380	-0.2067	0.3247	
11 - 2	-0.7147	-0.1833	0.3480	

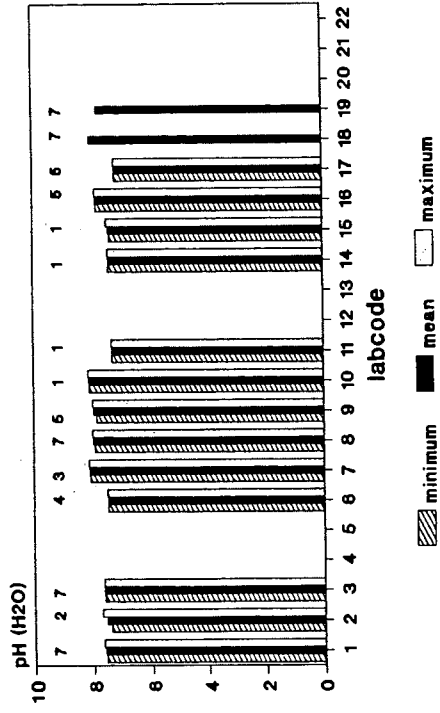
TABLE I : pH (H2O) - Overview of reported data

labcode	samples reported	number of replicates
1	A1 A2 B1 B2	3
2	A1 A2 B1 B2	3
3	A1 A2 B1 B2	5
4		
5	A2 B1	2
6	A1 A2 B1 B2	2
7	A1 A2 B1 B2	3
8	A1 A2 B1 B2	3
9	A1 A2 B1 B2	3
10	A1 A2 B1 B2	3
11	A1 A2 B1 B2	3
12		
13		
14	A1 A2 A3 B1 B2	3
15	A1 A2 B1 B2	3 B2:2
16	A1 A2 B1 B2	3
17	A1 A2 B1 B2	3
18	A1 A2 B1 B2	1 not reported
19	A1 A2 B1 B2	3
20		
21		
22		

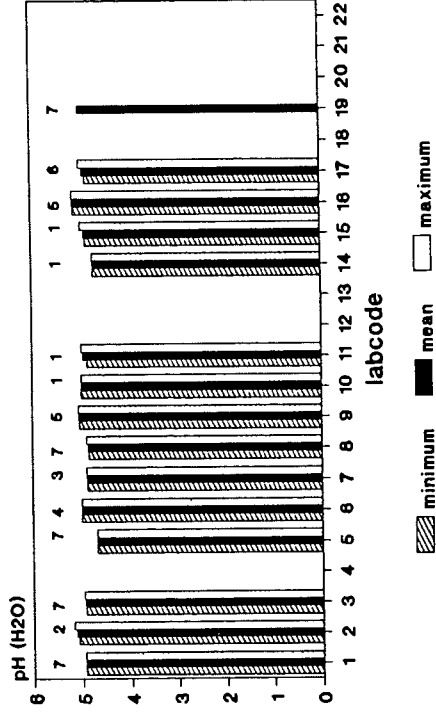
TABLE II : pH (H2O) - Summary method descriptions

method	description
1	1:5 soil:liquid, for H- & O-horizons 1:20
2	1:5 soil liquid, for H- & O-horizons 1:1
3	AFNOR X31-103
4	1:2 soil:liquid
5	1:2.5 soil:liquid
6	1:5 soil:liquid, for H- & O-horizons 1:25
7	unspecified

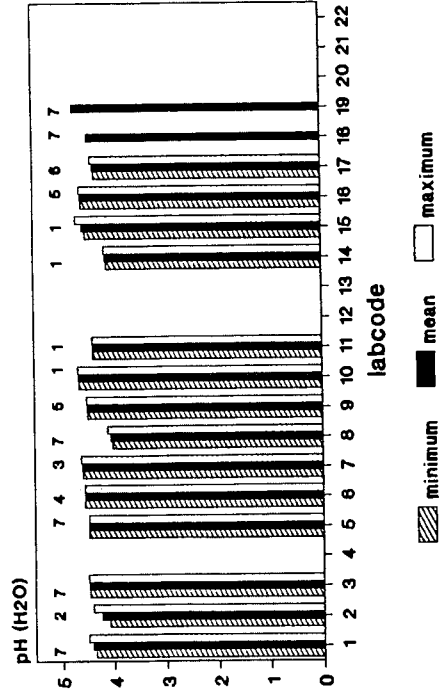
pH sample A1
national methods



pH sample A2
national methods



pH sample B1
national methods



pH sample B2
national methods

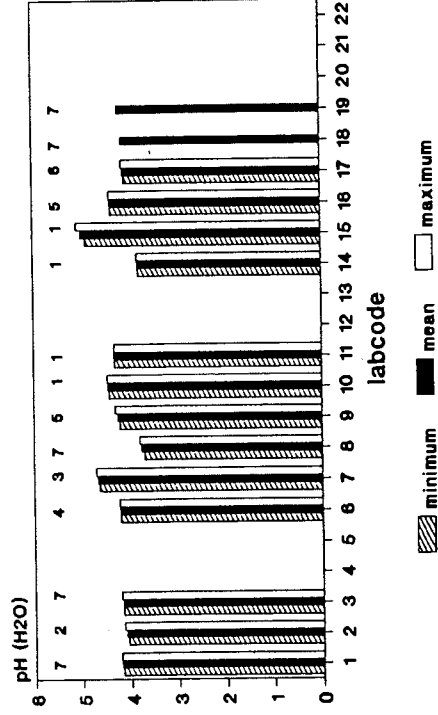


TABLE III : pH (H2O) - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	7.55	7.60	7.65	0.05	0.66
2	2	3	7.38	7.53	7.69	0.16	2.08
3	7	5	7.60	7.62	7.63	0.01	0.13
4							
5							
6	4	2	7.46	7.48	7.50	0.03	0.38
7	3	3	8.08	8.10	8.13	0.03	0.31
8	7	3	7.90	7.97	8.00	0.06	0.73
9	5	3	7.83	7.94	8.00	0.10	1.20
10	1	3	8.11	8.13	8.15	0.02	0.25
11	1	3	7.31	7.33	7.34	0.02	0.21
12							
13							
14	1	3	7.42	7.43	7.44	0.01	0.14
15	1	3	7.39	7.43	7.50	0.06	0.82
16	5	3	7.83	7.86	7.89	0.03	0.38
17	6	3	7.20	7.22	7.24	0.02	0.29
18	7	1		8.05			
19	7	3		7.80			
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	4.94	4.94	4.95	0.01	0.12
2	2	3	5.08	5.13	5.19	0.06	1.07
3	7	5	4.93	4.94	4.96	0.01	0.20
4							
5	7	1		4.68			
6	4	2	5.00	5.00	5.00	0.00	0.00
7	3	3	4.88	4.89	4.90	0.01	0.24
8	7	3	4.85	4.87	4.90	0.03	0.59
9	5	3	5.05	5.06	5.07	0.01	0.20
10	1	3	5.00	5.00	5.00	0.00	0.00
11	1	3	4.88	4.96	5.01	0.07	1.46
12							
13							
14	1	3	4.74	4.74	4.75	0.01	0.12
15	A3 1	3	4.69	4.70	4.71	0.01	0.21
16	1	3	4.90	4.93	5.00	0.06	1.17
17	5	3	5.15	5.16	5.17	0.01	0.22
18	6	3	4.89	4.95	5.02	0.07	1.35
19	7	3		5.02			
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	4.35	4.40	4.49	0.08	1.72
2	2	3	4.07	4.23	4.39	0.18	3.79
3	7	5	4.46	4.47	4.48	0.01	0.22
4							
5	7	1		4.47			
6	4	2	4.54	4.54	4.54	0.00	0.00
7	3	3	4.58	4.59	4.61	0.02	0.33
8	7	3	4.00	4.03	4.10	0.06	1.43
9	5	3	4.48	4.49	4.50	0.01	0.26
10	1	3	4.65	4.66	4.67	0.01	0.25
11	1	3	4.37	4.38	4.38	0.01	0.13
12							
13							
14	1	3	4.10	4.12	4.14	0.02	0.51
15	1	3	4.50	4.57	4.70	0.12	2.53
16	5	3	4.58	4.59	4.61	0.02	0.33
17	6	3	4.32	4.35	4.39	0.04	0.81
18	7	1		4.46			
19	7	3		4.73			
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	4.17	4.19	4.21	0.02	0.50
2	2	3	4.06	4.10	4.14	0.04	0.99
3	7	5	4.15	4.17	4.19	0.02	0.48
4							
5							
6	4	2	4.20	4.21	4.22	0.01	0.34
7	3	3	4.63	4.67	4.71	0.04	0.87
8	7	3	3.70	3.77	3.80	0.06	1.53
9	5	3	4.20	4.24	4.30	0.05	1.21
10	1	3	4.42	4.44	4.45	0.02	0.34
11	1	3	4.30	4.31	4.31	0.01	0.13
12							
13							
14	1	3	3.81	3.83	3.84	0.02	0.45
15	1	2	4.90	5.00	5.10	0.14	2.83
16	5	3	4.37	4.38	4.39	0.01	0.26
17	6	3	4.08	4.10	4.13	0.03	0.65
18	7	1		4.13			
19	7	3		4.21			
20							
21							
22							

TABLE V :

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Number of observations in by group = 38

Dependent Variable: PH(H2O)

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1.86005570	5.88	0.0003
Error	31	1.63429167		
Corrected Total	37	3.49434737		

PH(H2O) Mean

C.V.

R-Square 0.532304

2.987619

7.68526316

Source	DF	Type I SS	F Value	Pr > F
METH	6	1.86005570	5.88	0.0003
Source	DF	Type III SS	F Value	Pr > F
METH	6	1.86005570	5.88	0.0003

TABLE IV : pH (H2O) - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	12	7.58	0.34	0.70	0.00
A1	2	3	7.53	0.16	1.00	0.96
A1	3	3	8.10	0.03	0.99	0.78
A1	4	2	7.48	0.03	1.00	1.00
A1	5	6	7.90	0.08	0.83	0.10
A1	6	3	7.22	0.02	0.92	0.46
A1	7	9	7.80	0.20	0.89	0.19
A2	1	12	4.91	0.11	0.78	0.01
A2	2	3	5.13	0.06	1.00	0.90
A2	3	3	4.89	0.01	0.75	0.00
A2	4	2	5.00	0.00		
A2	5	6	5.11	0.06	0.80	0.06
A2	6	3	4.95	0.07	0.95	0.58
A2	7	9	4.90	0.10	0.86	0.09
A3	1	3	4.70	0.01	1.00	0.99
B1	1	12	4.43	0.22	0.90	0.14
B1	2	3	4.23	0.16	1.00	0.93
B1	3	3	4.59	0.02	0.96	0.64
B1	4	2	4.54	0.00		
B1	5	6	4.54	0.06	0.85	0.16
B1	6	3	4.35	0.04	0.99	0.84
B1	7	10	4.34	0.24	0.90	0.20
B2	1	11	4.34	0.41	0.90	0.19
B2	2	3	4.10	0.04	0.98	0.73
B2	3	3	4.67	0.04	0.98	0.73
B2	4	2	4.21	0.01	1.00	1.00
B2	5	6	4.31	0.08	0.87	0.23
B2	6	3	4.10	0.03	0.89	0.36
B2	7	9	4.04	0.21	0.75	0.01

Remark:

Labcode 3 and labcode 19 reported mean values and the number of replicates, respectively 5 and 3.
 To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.
 So, the "repl" values in this table can sometimes be different from the real number of replicates when these 2 laboratories are included.

SAMPLE=A1

General Linear Models Procedure

METH	Simultaneous		Difference	Simultaneous	
	Lower	Upper		Lower	Upper
METH	Confidence	Confidence		Confidence	Confidence
Comparison	Limit	Limit		Limit	Limit
				Means	

METH Comparison

2	-0.7135	-0.0533	0.6068
6	-0.3968	0.2633	0.9235
3	-1.4772	-0.8867	-0.2962
5	-1.1947	-0.6833	-0.1720
7	-1.0621	-0.5800	-0.0979
1	-0.8293	-0.3625	0.1043
2	-0.9072	-0.3167	0.2738
4	-0.9235	-0.2633	0.3968

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: PH(H2O)

NOTE. This test controls the type I experimentwise error rate.

alpha= 0.05 Confidence= 0.95 df= 31 MSE= 0.052719

Critical Value of Studentized Range= 4.454

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
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87					
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89					
90					
91					
92					
93					
94					
95					
96					
97					
98					
99					

Comparison

3	-	5	-0.3080	0.2033	0.7147	***
3	-	7	-0.1755	0.3067	0.7888	***
3	-	1	0.0573	0.5242	0.9910	***
3	-	2	-0.0205	0.5700	1.1605	***
3	-	4	-0.0368	0.6233	1.2835	***
3	-	6	0.2962	0.8867	1.4772	***
5	-	3	-0.7147	-0.2033	0.3080	***
5	-	7	-0.2778	0.1033	0.4845	***
5	-	1	-0.0408	0.3208	0.6824	***
5	-	2	-0.1447	0.3667	0.8780	***
5	-	4	-0.1705	0.4200	1.0105	***
5	-	6	0.1720	0.6833	1.1947	***
7	-	3	-0.7898	-0.3067	0.1755	***
7	-	5	-0.4845	-0.1033	0.2778	***
7	-	1	-0.1014	0.2175	0.5364	***
7	-	2	-0.2188	0.2633	0.7455	***
7	-	4	-0.2487	0.3167	0.8820	***
7	-	6	0.0979	0.5800	1.0621	***
1	-	3	-0.9910	-0.5242	-0.0573	***
1	-	5	-0.6824	-0.3208	0.0408	***
1	-	7	-0.5364	-0.2175	0.1014	***
1	-	2	-0.4210	0.0458	0.5127	***
1	-	4	-0.4532	0.0992	0.6515	***
1	-	6	-0.1043	0.3625	0.8293	***
2	-	3	-1.1605	-0.5700	0.0205	***
2	-	5	-0.8780	-0.3667	0.1447	***
2	-	7	-0.7455	-0.2633	0.2188	***
2	-	1	-0.5127	-0.0458	0.4210	***
2	-	4	-0.6068	0.0533	0.7135	***
2	-	6	-0.2738	0.3167	0.9072	***
4	-	3	-1.2835	-0.6233	0.0368	***
4	-	5	-1.0105	-0.4200	0.1705	***
4	-	7	-0.8820	-0.3167	0.2487	***
4	-	1	-0.6515	-0.0992	0.4532	***

pH (H2O) Method comparison

pH (H2O) Method comparison

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Tukey's Studentized Range (HSD) Test for variable: PH(H2O)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 31 MSE= 0.007796
Critical Value of Studentized Range= 4.454

Number of observations in by group = 39

Comparisons significant at the 0.05 level are indicated by '***'.

NOTE: Due to missing values, only 38 observations can be used in this analysis.

Dependent Variable: PH(H2O)

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.31394825	6.71	0.0001
Error	31	0.24168333		
Corrected Total	37	0.55563158		

R-Square	C.V.	PH(H2O) Mean
0.565030	1.779412	4.96210526

Source	DF	Type I SS	F Value	Pr > F
METH	6	0.31394825	6.71	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.31394825	6.71	0.0001

METH Comparison	Simultaneous Lower Confidence Limit	Simultaneous Upper Confidence Limit	Difference Between Means	
2 - 5	-0.1750	0.0217	0.0217	0.2183
2 - 4	-0.1205	0.1333	0.1333	0.3872
2 - 6	-0.0404	0.1867	0.1867	0.4137
2 - 1	0.0438	0.2233	0.2233	0.4029
2 - 7	0.0513	0.2367	0.2367	0.4221
2 - 3	0.0196	0.2467	0.2467	0.4737
5 - 2	-0.2183	-0.0217	-0.0217	0.1750
5 - 4	-0.1154	0.1117	0.1117	0.3387
5 - 6	-0.0317	0.1650	0.1650	0.3617
5 - 1	0.0626	0.2017	0.2017	0.3407
5 - 7	0.0684	0.2150	0.2150	0.3616
5 - 3	0.0283	0.2250	0.2250	0.4217
4 - 2	-0.3872	-0.1333	-0.1333	0.1205
4 - 5	-0.3387	-0.1117	-0.1117	0.1154
4 - 6	-0.2005	0.0533	0.0533	0.3072
4 - 1	-0.1224	0.0900	0.0900	0.3024
4 - 7	-0.1141	0.1033	0.1033	0.3207
4 - 3	-0.1405	0.1133	0.1133	0.3672
6 - 2	-0.4137	-0.1867	-0.1867	0.0404
6 - 5	-0.3617	-0.1650	-0.1650	0.0317
6 - 4	-0.3072	-0.0533	-0.0533	0.2005
6 - 1	-0.1429	0.0367	0.0367	0.2162
6 - 7	-0.1354	0.0500	0.0500	0.2354
6 - 3	-0.1671	0.0600	0.0600	0.2871
1 - 2	-0.4029	-0.2233	-0.2233	-0.0438
1 - 5	-0.3407	-0.2017	-0.2017	-0.0626
1 - 4	-0.3024	-0.0900	-0.0900	0.1224
1 - 6	-0.2162	-0.0367	-0.0367	0.1429
1 - 7	-0.1093	0.0133	0.0133	0.1360
1 - 3	-0.1562	0.0233	0.0233	0.2029
7 - 2	-0.4221	-0.2367	-0.2367	-0.0513
7 - 5	-0.3616	-0.2150	-0.2150	-0.0684
7 - 4	-0.3207	-0.1033	-0.1033	0.1141
7 - 6	-0.2354	-0.0500	-0.0500	0.1354

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Number of observations in by group = 39

Dependent Variable: PH(H2O)

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.39462731	1.89	0.1123
Error	32	1.11106500		
Corrected Total	38	1.50569231		

R-Square	C.V.	PH(H2O) Mean
0.262090	4.213527	4.42230769

Source	DF	Type I SS	F Value	Pr > F
METH	6	0.39462731	1.89	0.1123
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.39462731	1.89	0.1123

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 1	-0.1360	0.1093	-0.0133	0.1093	
7 - 3	-0.1754	0.1954	0.0100	0.1954	
3 - 2	-0.4737	-0.0196	-0.2467	-0.0196	***
3 - 5	-0.4217	-0.0283	-0.2250	-0.0283	***
3 - 4	-0.3672	0.1405	-0.1133	0.1405	
3 - 6	-0.2871	0.1671	-0.0600	0.1671	
3 - 1	-0.2029	0.1562	-0.0233	0.1562	
3 - 7	-0.1954	0.1754	-0.0100	0.1754	

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 6	-0.3949	-0.0093	-0.0093	-0.3949	0.3762
7 - 2	-0.2682	0.1173	0.1173	-0.2682	0.5029
2 - 3	-0.8449	-0.3667	-0.3667	-0.8449	0.1115
2 - 5	-0.7308	-0.3167	-0.3167	-0.7308	0.0975
2 - 4	-0.8480	-0.3133	-0.3133	-0.8480	0.2213
2 - 1	-0.5822	-0.2042	-0.2042	-0.5822	0.1739
2 - 6	-0.6049	-0.1267	-0.1267	-0.6049	0.3515
2 - 7	-0.5029	-0.1173	-0.1173	-0.5029	0.2682

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: PH(H2O)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 32 MSE= 0.034721
Critical Value of Studentized Range= 4.445

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
3 - 5	-0.3641	0.0500	0.0500	0.4641	0.4641
3 - 4	-0.4813	0.0533	0.0533	0.5880	0.5880
3 - 1	-0.2156	0.1625	0.1625	0.5406	0.5406
3 - 6	-0.2382	0.2400	0.2400	0.7182	0.7182
3 - 7	-0.1362	0.2493	0.2493	0.6349	0.6349
3 - 2	-0.1115	0.3667	0.3667	0.8449	0.8449
5 - 3	-0.4641	-0.0500	-0.0500	0.3641	0.3641
5 - 4	-0.4749	0.0033	0.0033	0.4815	0.4815
5 - 1	-0.1803	0.1125	0.1125	0.4053	0.4053
5 - 6	-0.2241	0.1900	0.1900	0.6041	0.6041
5 - 7	-0.1031	0.1993	0.1993	0.5018	0.5018
5 - 2	-0.0975	0.3167	0.3167	0.7308	0.7308
4 - 3	-0.5880	-0.0533	-0.0533	0.4813	0.4813
4 - 5	-0.4815	-0.0033	-0.0033	0.4749	0.4749
4 - 1	-0.3382	0.1092	0.1092	0.5565	0.5565
4 - 6	-0.3480	0.1867	0.1867	0.7213	0.7213
4 - 7	-0.2577	0.1960	0.1960	0.6497	0.6497
4 - 2	-0.2213	0.3133	0.3133	0.8480	0.8480
1 - 3	-0.5406	-0.1625	-0.1625	0.2156	0.2156
1 - 5	-0.4053	-0.1125	-0.1125	0.1803	0.1803
1 - 4	-0.5565	-0.1092	-0.1092	0.3382	0.3382
1 - 6	-0.3006	0.0775	0.0775	0.4556	0.4556
1 - 7	-0.1639	0.0868	0.0868	0.3376	0.3376
1 - 2	-0.1739	0.2042	0.2042	0.5822	0.5822
6 - 3	-0.7182	-0.2400	-0.2400	0.2382	0.2382
6 - 5	-0.6041	-0.1900	-0.1900	0.2241	0.2241
6 - 4	-0.7213	-0.1867	-0.1867	0.3480	0.3480
6 - 1	-0.4556	-0.0775	-0.0775	0.3006	0.3006
6 - 7	-0.3762	0.0093	0.0093	0.3949	0.3949
6 - 2	-0.3515	0.1267	0.1267	0.6049	0.6049
7 - 3	-0.6349	-0.2493	-0.2493	0.1362	0.1362
7 - 5	-0.5018	-0.1993	-0.1993	0.1031	0.1031
7 - 4	-0.6497	-0.1960	-0.1960	0.2577	0.2577
7 - 1	-0.3376	-0.0868	-0.0868	0.1639	0.1639

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: PH(H2O)

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 30 MSE= 0.07001
Critical Value of Studentized Range= 4.464

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Limit	Upper Limit		Lower Limit	Upper Limit
3	1	-0.216	0.328	0.328	0.873	0.873
3	5	-0.234	0.357	0.357	0.947	0.947
3	4	-0.306	0.457	0.457	1.219	1.219
3	2	-0.119	0.563	0.563	1.245	1.245
3	6	-0.115	0.567	0.567	1.249	1.249
3	7	0.067	0.623	0.623	1.180	1.180
					***	***
1	3	-0.873	-0.328	-0.328	0.216	0.216
1	5	-0.396	0.028	0.028	0.452	0.452
1	4	-0.514	0.128	0.128	0.770	0.770
1	2	-0.309	0.235	0.235	0.779	0.779
1	6	-0.306	0.238	0.238	0.782	0.782
1	7	-0.081	0.295	0.295	0.670	0.670
5	3	-0.947	-0.357	-0.357	0.234	0.234
5	1	-0.452	-0.028	-0.028	0.396	0.396
5	4	-0.582	0.100	0.100	0.782	0.782
5	2	-0.384	0.207	0.207	0.797	0.797
5	6	-0.381	0.210	0.210	0.801	0.801
5	7	-0.174	0.267	0.267	0.707	0.707
4	3	-1.219	-0.457	-0.457	0.306	0.306
4	1	-0.770	-0.128	-0.128	0.514	0.514
4	5	-0.782	-0.100	-0.100	0.582	0.582
4	2	-0.656	0.107	0.107	0.869	0.869
4	6	-0.652	0.110	0.110	0.872	0.872
4	7	-0.486	0.167	0.167	0.820	0.820
2	3	-1.245	-0.563	-0.563	0.119	0.119
2	1	-0.779	-0.235	-0.235	0.309	0.309
2	5	-0.797	-0.207	-0.207	0.384	0.384
2	4	-0.869	-0.107	-0.107	0.656	0.656
2	6	-0.679	0.003	0.003	0.685	0.685
2	7	-0.497	0.060	0.060	0.617	0.617
6	3	-1.249	-0.567	-0.567	0.115	0.115
6	1	-0.782	-0.238	-0.238	0.306	0.306
6	5	-0.801	-0.210	-0.210	0.381	0.381
6	4	-0.872	-0.110	-0.110	0.652	0.652

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Number of observations in by group = 37

Dependent Variable: PH(H2O)

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1.14591384	2.73	0.0310
Error	30	2.10029697		
Corrected Total	36	3.24621081		
R-Square		C.V.		PH(H2O) Mean
	0.353000	6.235651		4.24324324

Source	DF	Type I SS	F Value	Pr > F
METH	6	1.14591384	2.73	0.0310
Source	DF	Type III SS	F Value	Pr > F
METH	6	1.14591384	2.73	0.0310

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
6 - 2	-0.685	-0.003	0.679	
6 - 7	-0.500	0.057	0.613	
7 - 3	-1.180	-0.623	-0.067	***
7 - 1	-0.670	-0.295	0.081	
7 - 5	-0.707	-0.267	0.174	
7 - 4	-0.820	-0.167	0.486	
7 - 2	-0.617	-0.060	0.497	
7 - 6	-0.613	-0.057	0.500	

3. ORGANIC C

units: g/kg
mandatory for all samples
reported by 20 laboratories

TABLE I : Organic C - Overview of the reported data

labcode	samples reported	number of replicates	N/R method
1	A1 A2 B1 B2	3	N A2: N/R
2	A1 A2 B1 B2	3	N
3			
4	A1 A2 B1 B2	4	R
5	A2 B1 B2	1	R
6	A1 A2 B1 B2	2	R A2: N/R
7	A1 A2 B1 B2	3	N/R
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N=R
10	A1 A2 B1 B2	3	N
11	A1 A2 B1 B2	3	N
12	A1 A2 B1 B2	5	N
13	A1 A2 B1 B2	3	R
14	A1 A2 B1 B2	5	N
15	A1 A2 B1 B2	2	N
16	A1 A2 B1 B2	3	N
17	A1 A2 B1 B2	3	N=R
18	A1 A2 B1 B2	2	N=R
19	A1 A2 B1 B2	3	N
20	A1 A2 B1 B2	1 not reported	N
21	A1 A2 B1 B2	3	N
22	A1 A2 B1 B2	3	R

TABLE II : Organic C - Summary method descriptions

method	description
1	reference
2	wet ashing
3	Walkley & Black
4	ignition (dry combustion)
5	AFNOR X31-109
6	Tjurin (wet combustion)
7	ignition
8	thermal combustion
9	titration
10	LECO
11	unspecified

TABLE IIIa : Organic C - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	20.50	20.87	21.50	0.55	2.64
2	3	3	22.10	22.97	23.50	0.76	3.30
3							
4							
5							
6							
7	5	3	21.70	22.57	23.30	0.81	3.58
8	3	3	18.80	19.67	20.10	0.75	3.82
9							
10	6	3	18.40	19.27	19.90	0.78	4.03
11	11	3	24.00	25.00	26.00	1.00	4.00
12	7	5	24.50	27.30	28.30	1.62	5.93
13							
14	10	5	23.48	24.76	26.28	1.24	5.02
15	3	2	23.50	24.50	25.50	1.41	5.77
16	8	3	54.40	54.70	55.20	0.44	0.80
17							
18							
19	11	1		20.50			
20	11	1		0.18			
21	9	3	21.50	21.60	21.70	0.10	0.46
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	291.00	304.93	313.80	12.22	4.01
2	3	3	292.80	317.20	331.60	21.25	6.70
3							
4							
5							
6	4	2	768.60	789.20	809.80	29.13	3.69
7	5	3	406.70	412.30	420.20	7.04	1.71
8	3	3	771.80	775.43	779.40	3.81	0.49
9							
10	6	3	333.80	341.10	345.90	6.43	1.88
11	11	3	460.00	466.87	470.00	5.77	1.24
12	7	5	441.40	445.86	451.70	3.76	0.84
13							
14	10	5	381.40	387.05	393.40	4.94	1.28
14 A3	10	5	386.60	415.03	423.60	14.16	3.41
15	3	2	286.20	287.90	289.60	2.40	0.84
16	8	3	428.20	434.93	438.70	5.85	1.34
17							
18							
19	11	1		466.70			
20	11	1		40.10			
21	9	3	417.90	419.97	423.00	2.68	0.64
22							

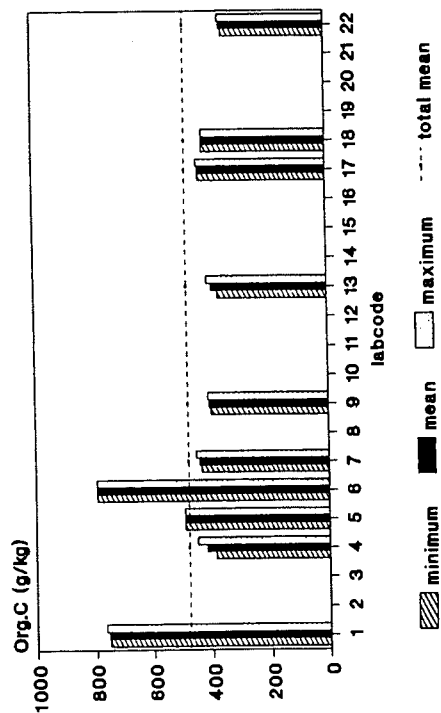
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	1.20	1.23	1.30	0.06	4.88
2	3	3	1.70	1.90	2.30	0.35	18.23
3							
4							
5							
6							
7	5	3	1.80	2.07	2.40	0.31	14.78
8	3	3	2.00	2.67	3.00	0.58	21.65
9							
10	6	3	1.70	1.70	1.70	0.00	0.00
11	11	3	6.00	6.33	7.00	0.58	9.12
12	7	5	11.60	12.22	13.50	0.77	6.27
13							
14	10	5	1.40	1.46	1.60	0.09	6.13
15	3	2	1.02	1.53	2.04	0.72	47.14
16	8	3	1.50	1.50	1.50	0.00	0.00
17							
18							
19	11	1		1.70			
20	11	1		0.29			
21	8	3	3.30	3.60	3.90	0.30	8.33
22							

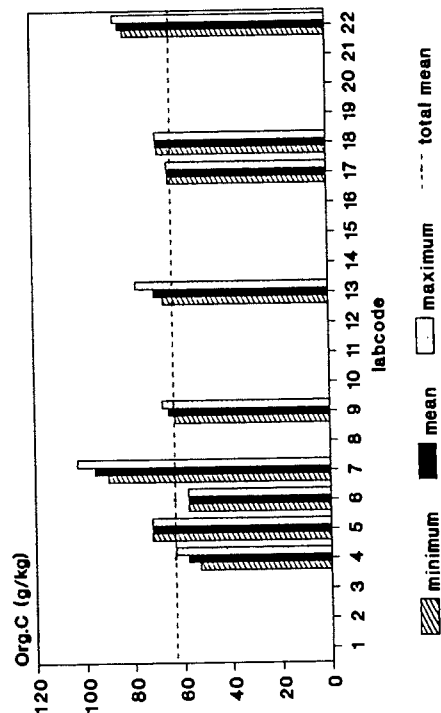
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	49.20	52.87	55.10	3.08	5.85
2	3	3	64.40	70.93	74.70	5.68	8.01
3							
4							
5							
6							
7	5	3	90.50	93.63	96.10	2.86	3.05
8	3	3	6.30	43.85	63.46	32.53	74.19
9							
10	6	3	69.60	69.90	70.50	0.52	0.74
11	11	3	65.00	66.00	67.00	1.00	1.52
12	7	5	75.60	77.38	78.30	1.05	1.35
13							
14	10	5	74.60	79.96	88.80	5.58	6.98
15	3	2	108.10	111.15	114.20	4.31	3.88
16	8	3	72.80	77.20	82.90	5.17	6.70
17							
18							
19	11	1		65.50			
20	11	1		6.38			
21	8	3	81.00	82.53	83.60	1.36	1.65
22							

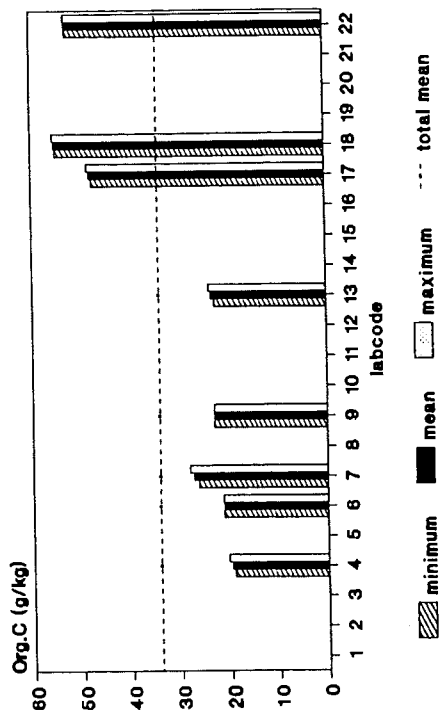
Org.C sample A2
reference method



Org.C sample B2
reference method



Org.C sample A1
reference method



Org.C sample B1
reference method

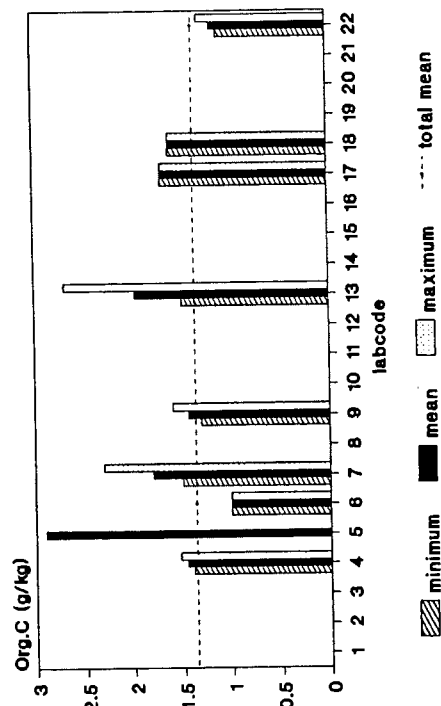


TABLE IIIb : Organic carbon - reference method

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	19.21	19.77	20.32	0.79	3.97
5						
6	2	21.10	21.20	21.30	0.14	0.67
7	3	26.20	27.13	27.90	0.86	3.18
8						
9	3	23.00	23.00	23.00	0.00	0.00
10						
11						
12						
13	3	22.80	23.53	24.00	0.64	2.73
14						
15						
16						
17	3	47.70	48.13	48.60	0.45	0.94
18	2	55.17	55.38	55.58	0.29	0.52
19						
20						
21						
22	3	52.80	52.93	53.10	0.15	0.29

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1	3	751.80	757.13	763.80	6.11	0.81
2						
3						
4	2	385.90	417.00	448.10	43.98	10.55
5	1		491.00			
6	2	792.50	793.90	795.30	1.98	0.25
7	3	430.70	440.97	451.60	10.46	2.37
8						
9	3	398.00	403.33	408.00	5.03	1.25
10						
11						
12						
13	3	371.70	392.17	408.90	18.88	4.81
14						
15						
16						
17	3	430.90	433.63	437.60	3.43	0.79
18	2	419.58	419.58	419.58	0.00	0.00
19						
20						
21						
22	3	350.20	355.67	361.70	5.77	1.62

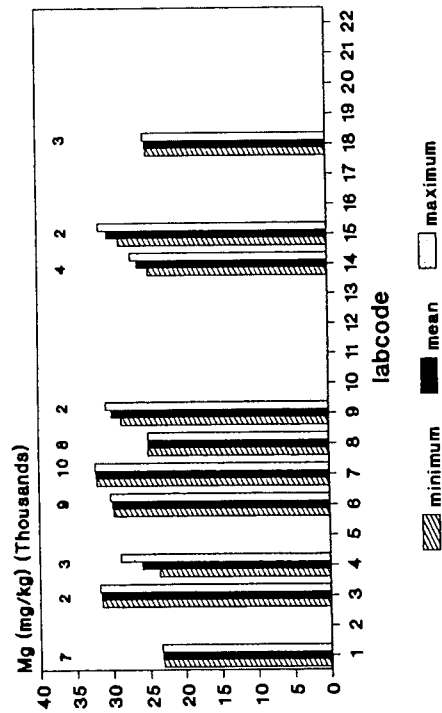
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	1.39	1.46	1.53	0.10	6.78
5	1		2.90			
6	2	1.00	1.00	1.00	0.00	0.00
7	3	1.50	1.80	2.30	0.44	24.22
8						
9	3	1.30	1.43	1.60	0.15	10.66
10						
11						
12						
13	3	1.50	1.97	2.70	0.64	32.69
14						
15						
16						
17	3	1.70	1.70	1.70	0.00	0.00
18	2	1.62	1.62	1.62	0.00	0.00
19						
20						
21						
22	3	1.10	1.17	1.30	0.12	9.90

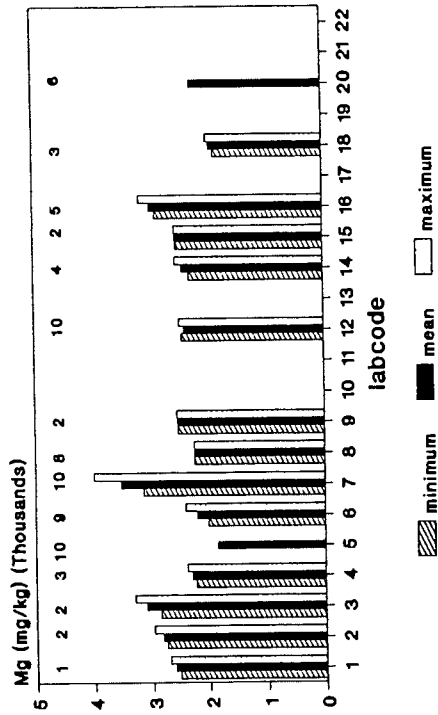
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	53.10	58.15	63.20	7.14	12.28
5	1		72.60			
6	2	57.90	58.00	58.10	0.14	0.24
7	3	90.20	95.97	103.30	6.69	6.97
8						
9	3	83.00	85.67	88.00	2.52	3.83
10						
11						
12						
13	3	87.10	70.93	78.10	6.21	8.76
14						
15						
16						
17	3	64.70	64.90	65.20	0.27	0.41
18	2	69.02	69.38	69.73	0.50	0.72
19						
20						
21						
22	3	82.10	84.20	86.10	2.01	2.38

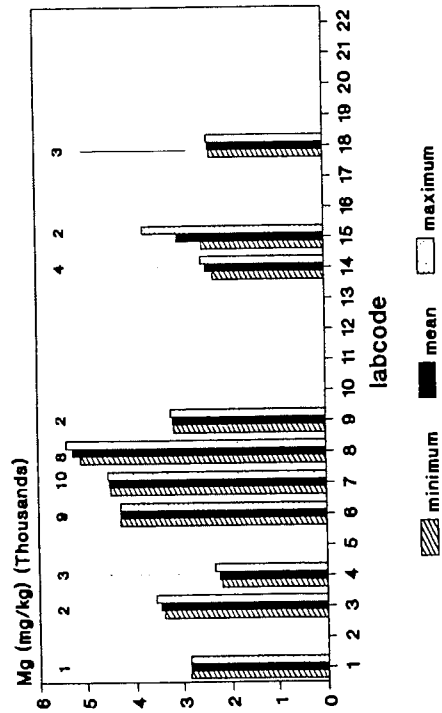
Mg sample A1
national methods



Mg sample A2
national methods



Mg sample B1
national methods



Mg sample B2
national methods

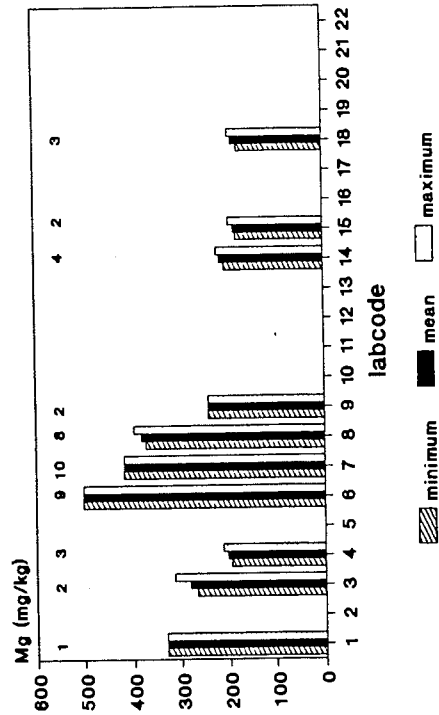


TABLE V : OC Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 5 6 7 8 9

Number of observations in by group = 62

NOTE: Due to missing values, only 59 observations can be used in this analysis.

Dependent Variable: OC

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	4243.93419973	4.94	0.0001
Error	49	4679.94349857		
Corrected Total	58	8923.87769831		

R-Square C.V. OC Mean

0.475571 34.59403 28.25016949

Source	DF	Type I SS	F Value	Pr > F
METH	9	4243.93419973	4.94	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	4243.93419973	4.94	0.0001

TABLE IV : Organic C - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	21	34.14	14.42	0.78	0.00
A1	2	3	20.87	0.55	0.82	0.17
A1	3	8	22.11	2.26	0.94	0.59
A1	5	3	22.57	0.81	0.98	0.73
A1	6	3	19.27	0.78	0.93	0.50
A1	7	5	27.30	1.62	0.73	0.02
A1	8	3	54.70	0.44	0.84	0.22
A1	9	3	21.60	0.10	1.00	0.99
A1	10	5	24.76	1.24	0.91	0.44
A1	11	5	19.14	10.80	0.71	0.02
A2	1	27	477.36	226.11	0.86	0.00
A2	2	3	304.93	12.22	0.87	0.30
A2	3	8	481.71	243.80	0.69	0.00
A2	4	2	789.20	29.13	1.00	1.00
A2	5	3	412.30	7.04	0.92	0.45
A2	6	3	341.10	6.43	0.89	0.34
A2	7	5	445.86	3.76	0.94	0.70
A2	8	3	434.93	5.85	0.81	0.13
A2	9	3	419.97	2.68	0.90	0.39
A2	10	4	387.05	4.94	0.98	0.84
A2	11	5	381.36	190.81	0.57	0.00
A3	10	6	415.03	14.16	0.66	0.00
B1	1	22	1.36	0.63	0.91	0.05
B1	2	3	1.23	0.06	0.75	0.00
B1	3	8	2.10	0.67	0.93	0.55
B1	5	3	2.07	0.31	0.96	0.84
B1	6	3	1.70	0.00	0.00	0.00
B1	7	5	12.22	0.77	0.84	0.17
B1	8	3	1.50	0.00	1.00	0.99
B1	9	3	3.60	0.30	0.77	0.05
B1	10	5	1.46	0.09	0.84	0.17
B1	11	5	4.20	2.99	0.84	0.00
B2	1	22	63.54	26.23	0.84	0.00
B2	2	3	52.67	3.08	0.92	0.44
B2	3	8	70.83	33.03	0.89	0.23
B2	5	3	93.63	2.86	0.96	0.61
B2	6	3	69.90	0.52	0.75	0.00
B2	7	5	77.38	1.05	0.84	0.15
B2	8	3	77.20	5.17	0.95	0.58
B2	9	3	82.53	1.36	0.91	0.42
B2	10	5	79.96	5.58	0.91	0.49
B2	11	5	53.98	26.62	0.58	0.00

Remark:
Labcode 19 reported the mean values of 3 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values for method 11 in this table are different from the real number
of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: OC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 49 MSE= 95.50905
Critical Value of Studentized Range= 4.686

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Difference Between Means	Simultaneous		
	Lower Confidence Limit		Upper Confidence Limit		Lower Confidence Limit		Upper Confidence Limit
8 - 1	0.577	20.563	40.549	***			
8 - 7	3.752	27.400	51.048	***			
8 - 10	6.292	29.940	53.588	***			
8 - 5	5.694	32.133	58.573	***			
8 - 3	10.665	32.588	54.510	***			
8 - 9	6.661	33.100	59.539	***			
8 - 2	7.394	33.833	60.273	***			
8 - 6	8.994	35.433	61.873	***			
8 - 11	11.916	35.564	59.212	***			
1 - 8	-40.549	-20.563	-0.577	***			
1 - 7	-9.276	6.837	22.950				
1 - 10	-6.736	9.377	25.490				
1 - 5	-8.416	11.570	31.557				
1 - 3	-1.429	12.025	25.478				
1 - 9	-7.449	12.537	32.523				
1 - 2	-6.716	13.270	33.257				
1 - 6	-5.116	14.870	34.857				
1 - 11	-1.112	15.001	31.114				
7 - 8	-51.048	-27.400	-3.752	***			
7 - 1	-22.950	-6.837	9.276				
7 - 10	-17.940	2.540	23.020				
7 - 5	-18.915	4.733	28.381				
7 - 3	-13.273	5.187	23.648				
7 - 9	-17.948	5.700	29.348				
7 - 2	-17.215	6.433	30.081				
7 - 6	-15.615	8.033	31.681				
7 - 11	-12.316	8.164	28.644				
10 - 8	-53.588	-29.940	-6.292	***			
10 - 1	-25.490	-9.377	6.736				
10 - 7	-23.020	-2.540	17.940				
10 - 5	-21.455	2.193	25.841				
10 - 3	-15.813	2.648	21.108				
10 - 9	-20.488	3.160	26.808				
10 - 2	-19.755	3.893	27.541				
10 - 6	-18.155	5.493	29.141				
10 - 11	-14.856	5.624	26.104				

SAMPLE=A1

SAMPLE=A1

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	11	1 10 11 2 3 4 5 6 7 8 9

Number of observations in by group = 66

Dependent Variable: OC

Source	DF	Sum of Squares	F Value	Pr > F
Model	10	404199.772336	1.17	0.3276
Error	55	1892543.970350		
Corrected Total	65	2296743.742686		

R-Square C.V. OC Mean

0.175988 41.15977 450.68045455

Source	DF	Type I SS	F Value	Pr > F
METH	10	404199.772336	1.17	0.3276
Source	DF	Type III SS	F Value	Pr > F
METH	10	404199.772336	1.17	0.3276

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous Confidence Limit		Difference Between Means	Simultaneous Upper Confidence Limit	
	Lower Limit	Upper Limit		Lower Limit	Upper Limit
6 - 11	-23.517	23.779	0.131	-11.916	***
11 - 8	-59.212	-35.564	-15.001	12.316	
11 - 1	-31.114	-8.164	-5.624	20.217	
11 - 7	-28.644	-3.431	-2.976	15.484	
11 - 10	-26.104	-2.464	-1.731	21.184	
11 - 5	-27.079	-0.131		21.917	
11 - 3	-21.437			23.517	
11 - 9	-26.112				
11 - 2	-25.379				
11 - 6	-23.779				

----- SAMPLE=A2 -----

General Linear Models Procedure

General Linear Models Procedure				General Linear Models Procedure			
METH	Comparison	Simultaneous		METH	Comparison	Simultaneous	
		Lower Confidence Limit	Difference Between Means			Lower Confidence Limit	Upper Confidence Limit
7	- 10	-359.00	58.81	7	- 10	-359.00	476.62
7	- 11	-329.42	64.50	7	- 11	-329.42	458.42
7	- 6	-350.09	104.76	7	- 6	-350.09	559.61
7	- 2	-313.93	140.93	7	- 2	-313.93	595.78
8	- 4	-922.84	-354.27	8	- 4	-922.84	214.30
8	- 3	-468.44	-46.78	8	- 3	-468.44	374.88
8	- 1	-421.48	-42.43	8	- 1	-421.48	336.62
8	- 7	-465.78	-10.93	8	- 7	-465.78	443.93
8	- 9	-493.58	14.97	8	- 9	-493.58	523.51
8	- 5	-485.91	22.63	8	- 5	-485.91	531.18
8	- 10	-427.82	47.88	8	- 10	-427.82	523.58
8	- 11	-401.28	53.57	8	- 11	-401.28	508.43
8	- 6	-414.71	93.83	8	- 6	-414.71	602.38
8	- 2	-378.54	130.00	8	- 2	-378.54	638.54
9	- 4	-937.80	-369.23	9	- 4	-937.80	199.34
9	- 3	-483.41	-61.75	9	- 3	-483.41	359.92
9	- 1	-436.44	-57.40	9	- 1	-436.44	321.65
9	- 7	-480.75	-25.89	9	- 7	-480.75	428.96
9	- 8	-523.51	-14.97	9	- 8	-523.51	493.58
9	- 5	-500.88	7.67	9	- 5	-500.88	516.21
9	- 10	-442.78	32.92	9	- 10	-442.78	508.62
9	- 11	-416.25	38.61	9	- 11	-416.25	493.46
9	- 6	-429.68	78.87	9	- 6	-429.68	587.41
9	- 2	-393.51	115.03	9	- 2	-393.51	623.58
5	- 4	-945.47	-376.90	5	- 4	-945.47	191.67
5	- 3	-491.07	-69.41	5	- 3	-491.07	332.25
5	- 1	-444.11	-65.06	5	- 1	-444.11	313.98
5	- 7	-488.41	-33.56	5	- 7	-488.41	421.29
5	- 8	-531.18	-22.63	5	- 8	-531.18	485.91
5	- 9	-516.21	-7.67	5	- 9	-516.21	500.88
5	- 10	-450.45	25.25	5	- 10	-450.45	500.95
5	- 11	-423.91	30.94	5	- 11	-423.91	485.79
5	- 6	-437.34	71.20	5	- 6	-437.34	579.74
5	- 2	-401.18	107.37	5	- 2	-401.18	615.91
10	- 4	-941.54	-402.15	10	- 4	-941.54	137.24
10	- 3	-476.07	-94.66	10	- 3	-476.07	286.74
10	- 1	-424.00	-90.31	10	- 1	-424.00	243.38
10	- 7	-476.62	-58.81	10	- 7	-476.62	359.00
10	- 8	-523.58	-47.88	10	- 8	-523.58	427.82
10	- 9	-508.62	-32.92	10	- 9	-508.62	442.78
10	- 5	-500.95	-25.25	10	- 5	-500.95	450.45
10	- 11	-412.12	5.69	10	- 11	-412.12	423.50
10	- 6	-429.75	45.95	10	- 6	-429.75	521.65
10	- 2	-393.58	82.12	10	- 2	-393.58	557.82

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: OC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 55 MSE= 34409.89
Critical value of Studentized Range= 4.748

Comparisons significant at the 0.05 level are indicated by '***'.

General Linear Models Procedure				General Linear Models Procedure			
METH	Comparison	Simultaneous		METH	Comparison	Simultaneous	
		Lower Confidence Limit	Difference Between Means			Lower Confidence Limit	Upper Confidence Limit
4	- 3	-184.91	307.49	4	- 3	-184.91	799.88
4	- 1	-144.59	311.84	4	- 1	-144.59	768.27
4	- 7	-177.76	343.34	4	- 7	-177.76	864.44
4	- 8	-214.30	354.27	4	- 8	-214.30	922.84
4	- 9	-199.34	369.23	4	- 9	-199.34	937.80
4	- 5	-191.67	376.90	4	- 5	-191.67	945.47
4	- 10	-137.24	402.15	4	- 10	-137.24	941.54
4	- 11	-113.26	407.84	4	- 11	-113.26	928.94
4	- 6	-120.47	448.10	4	- 6	-120.47	1016.67
4	- 2	-84.30	484.27	4	- 2	-84.30	1052.84
3	- 4	-799.88	-307.49	3	- 4	-799.88	184.91
3	- 1	-246.37	4.35	3	- 1	-246.37	255.06
3	- 7	-319.22	35.85	3	- 7	-319.22	390.92
3	- 8	-374.88	46.78	3	- 8	-374.88	468.44
3	- 9	-359.92	61.75	3	- 9	-359.92	483.41
3	- 5	-352.25	69.41	3	- 5	-352.25	491.07
3	- 10	-286.74	94.66	3	- 10	-286.74	476.07
3	- 11	-254.72	100.35	3	- 11	-254.72	455.42
3	- 6	-281.05	140.61	3	- 6	-281.05	562.27
3	- 2	-244.88	176.78	3	- 2	-244.88	598.44
1	- 4	-768.27	-311.84	1	- 4	-768.27	144.59
1	- 3	-255.06	-4.35	1	- 3	-255.06	246.37
1	- 7	-271.73	31.50	1	- 7	-271.73	334.74
1	- 8	-336.62	42.43	1	- 8	-336.62	421.48
1	- 9	-321.65	57.40	1	- 9	-321.65	436.44
1	- 5	-313.98	65.06	1	- 5	-313.98	444.11
1	- 10	-243.38	90.31	1	- 10	-243.38	424.00
1	- 11	-207.23	96.00	1	- 11	-207.23	399.24
1	- 6	-242.78	136.26	1	- 6	-242.78	515.31
1	- 2	-206.62	172.43	1	- 2	-206.62	551.48
7	- 4	-864.44	-343.34	7	- 4	-864.44	177.76
7	- 3	-390.92	-35.85	7	- 3	-390.92	319.22
7	- 1	-334.74	-31.50	7	- 1	-334.74	271.73
7	- 8	-443.93	10.93	7	- 8	-443.93	465.78
7	- 9	-428.96	25.89	7	- 9	-428.96	480.75
7	- 5	-421.29	33.56	7	- 5	-421.29	488.41

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 5 6 7 8 9

Number of observations in by group = 60

Dependent Variable: OC

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	531.49506091	58.88	0.0001
Error	50	50.14447242		

Corrected Total

R-Square

C.V.

OC Mean

0.913788

36.10975

2.77333333

Source

DF

F Value

Pr > F

METH

9

58.88

0.0001

Source

DF

F Value

Pr > F

METH

9

58.88

0.0001

General Linear Models Procedure

Simultaneous

Lower

Confidence

Limit

METH

Comparison

11

- 4

- 3

- 1

- 7

- 8

- 9

- 5

- 10

- 6

- 2

6

- 4

- 3

- 1

- 7

- 8

- 9

- 5

- 10

- 11

- 2

6

2

- 4

- 3

- 1

- 7

- 8

- 9

- 5

- 10

- 11

- 6

2

2

2

2

2

2

2

2

2

2

Difference

Between

Means

-407.84

-100.35

-96.00

-64.50

-53.57

-38.61

-30.94

-5.69

40.26

76.43

-448.10

-140.61

-136.26

-104.76

-93.83

-78.87

-71.20

-45.95

-40.26

36.17

-484.27

-176.78

-172.43

-140.93

-130.00

-115.03

-107.37

-82.12

-76.43

-36.17

84.30

244.88

206.62

313.93

378.54

393.51

401.18

393.58

378.43

472.38

472.38

472.38

472.38

472.38

472.38

472.38

472.38

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: OC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 50 MSE= 1.002889
Critical Value of Studentized Range= 4.682

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous Lower Confidence Limit
Simultaneous Upper Confidence Limit
Difference Between Means
METH Comparison

Simultaneous Lower Confidence Limit
Simultaneous Upper Confidence Limit
Difference Between Means
METH Comparison

7	- 11	5.925	8.022	10.119	***
7	- 9	6.199	8.620	11.041	***
7	- 3	8.235	10.125	12.015	***
7	- 5	7.732	10.153	12.574	***
7	- 6	8.099	10.520	12.941	***
7	- 8	8.299	10.720	13.141	***
7	- 10	8.663	10.760	12.857	***
7	- 1	9.216	10.859	12.501	***
7	- 2	8.566	10.987	13.408	***
11	- 7	-10.119	-8.022	-5.925	***
11	- 9	-1.823	0.598	3.019	***
11	- 3	0.213	2.103	3.993	***
11	- 5	-0.290	2.131	4.552	***
11	- 6	0.077	2.498	4.919	***
11	- 8	0.277	2.698	5.119	***
11	- 10	0.641	2.738	4.835	***
11	- 1	1.194	2.837	4.479	***
11	- 2	0.544	2.965	5.386	***
9	- 7	-11.041	-8.620	-6.199	***
9	- 11	-3.019	-0.598	1.823	***
9	- 3	-0.739	1.505	3.749	***
9	- 5	-1.173	1.533	4.240	***
9	- 6	-0.807	1.900	4.607	***
9	- 8	-0.607	2.100	4.807	***
9	- 10	-0.281	2.140	4.561	***
9	- 1	0.198	2.239	4.279	***
9	- 2	-0.340	2.367	5.073	***
3	- 7	-12.015	-10.125	-8.235	***
3	- 11	-3.993	-2.103	-0.213	***
3	- 9	-3.749	-1.505	0.739	***
3	- 5	-2.216	0.028	2.273	***
3	- 6	-1.849	0.395	2.639	***
3	- 8	-1.649	0.595	2.839	***
3	- 10	-1.255	0.635	2.525	***
3	- 1	-0.635	0.734	2.102	***
3	- 2	-1.383	0.862	3.106	***

Simultaneous Lower Confidence Limit
Simultaneous Upper Confidence Limit
Difference Between Means
METH Comparison

5	- 7	-12.574	-10.153	-7.732	***
5	- 11	-4.552	-2.131	0.290	***
5	- 9	-4.240	-1.533	1.173	***
5	- 3	-2.273	-0.028	2.216	***
5	- 6	-2.340	0.367	3.073	***
5	- 8	-2.140	0.567	3.273	***
5	- 10	-1.814	0.607	3.028	***
5	- 1	-1.335	0.705	2.746	***
5	- 2	-1.873	0.833	3.540	***
6	- 7	-12.941	-10.520	-8.099	***
6	- 11	-4.919	-2.498	-0.077	***
6	- 9	-4.607	-1.900	0.807	***
6	- 3	-2.639	-0.395	1.849	***
6	- 5	-3.073	-0.367	2.340	***
6	- 8	-2.507	0.200	2.907	***
6	- 10	-2.181	0.240	2.661	***
6	- 1	-1.702	0.339	2.379	***
6	- 2	-2.240	0.467	3.173	***
8	- 7	-13.141	-10.720	-8.299	***
8	- 11	-5.119	-2.698	-0.277	***
8	- 9	-4.807	-2.100	0.607	***
8	- 3	-2.839	-0.595	1.649	***
8	- 5	-3.273	-0.567	2.140	***
8	- 8	-2.907	-0.200	2.507	***
8	- 6	-2.381	0.040	2.461	***
8	- 10	-1.902	0.139	2.179	***
8	- 1	-2.440	0.267	2.973	***
10	- 7	-12.857	-10.760	-8.663	***
10	- 11	-4.835	-2.738	-0.641	***
10	- 9	-4.561	-2.140	0.281	***
10	- 3	-2.525	-0.635	1.255	***
10	- 5	-3.028	-0.607	1.814	***
10	- 8	-2.661	-0.240	2.181	***
10	- 6	-2.461	-0.040	2.381	***
10	- 10	-1.544	0.099	1.741	***
10	- 1	-2.194	0.227	2.648	***
1	- 7	-12.501	-10.859	-9.216	***
1	- 11	-4.479	-2.837	-1.194	***
1	- 9	-4.279	-2.239	-0.198	***
1	- 3	-2.102	-0.734	0.635	***
1	- 5	-2.746	-0.705	1.335	***
1	- 8	-2.379	-0.339	1.702	***
1	- 6	-2.179	-0.139	1.902	***
1	- 10	-1.741	-0.099	1.544	***

SAMPLE=B1

General Linear Models Procedure

METH Comparison		Simultaneous		Simultaneous Upper Confidence Limit
		Lower Confidence Limit	Difference Between Means	
1	- 2	-1.912	0.128	2.168
2	- 7	-13.408	-10.987	-8.566 ***
2	- 11	-5.386	-2.965	-0.544 ***
2	- 9	-5.073	-2.367	0.340
2	- 3	-3.106	-0.862	1.383
2	- 5	-3.540	-0.833	1.873
2	- 6	-3.173	-0.467	2.240
2	- 8	-2.973	-0.267	2.440
2	- 10	-2.648	-0.227	2.194
2	- 1	-2.168	-0.128	1.912

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 2 3 5 6 7 8 9

Number of observations in by group = 60

Dependent Variable: OC

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	6136.51516273	1.36	0.2334
Error	50	25143.76019727		
Corrected Total	59	31280.27536000		

R-Square	C.V.	OC Mean
0.196178	32.43026	69.14800000

Source	DF	Type I SS	F Value	Pr > F
METH	9	6136.51516273	1.36	0.2334
Source	DF	Type III SS	F Value	Pr > F
METH	9	6136.51516273	1.36	0.2334

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
8 - 5	-77.045	-16.433	44.178
8 - 9	-65.945	-5.333	55.278
8 - 10	-56.973	-2.760	51.453
8 - 7	-54.393	-0.180	54.033
8 - 3	-43.889	6.367	56.624
8 - 6	-53.312	7.300	67.912
8 - 1	-32.026	13.662	59.350
8 - 11	-30.989	23.224	77.437
8 - 2	-36.078	24.533	85.145
3 - 5	-73.057	-22.801	27.456
3 - 9	-61.957	-11.701	38.556
3 - 10	-51.447	-9.127	33.192
3 - 7	-48.867	-6.547	35.772
3 - 8	-56.624	-6.367	43.889
3 - 6	-49.324	0.933	51.189
3 - 1	-23.354	7.294	37.943
3 - 11	-25.463	16.857	59.176
3 - 2	-32.091	18.166	68.422
6 - 5	-84.345	-23.733	36.878
6 - 9	-73.245	-12.633	47.978
6 - 10	-64.273	-10.060	44.153
6 - 7	-61.693	-7.480	46.733
6 - 8	-67.912	-7.300	53.312
6 - 3	-51.189	-0.933	49.324
6 - 1	-39.326	6.362	52.050
6 - 11	-38.289	15.924	70.137
6 - 2	-43.378	17.233	77.845
1 - 5	-75.783	-30.095	15.593
1 - 9	-64.683	-18.995	26.693
1 - 10	-53.200	-16.422	20.356
1 - 7	-50.620	-13.842	22.936
1 - 8	-59.350	-13.662	32.026
1 - 3	-37.943	-7.294	23.354
1 - 6	-52.050	-6.362	39.326
1 - 11	-27.216	9.562	46.340
1 - 2	-34.816	10.872	56.559
11 - 5	-93.870	-39.657	14.555
11 - 9	-82.770	-28.557	25.655
11 - 10	-72.934	-25.984	20.966
11 - 7	-70.354	-23.404	23.546
11 - 8	-77.437	-23.224	30.989
11 - 3	-59.176	-16.857	25.463
11 - 6	-70.137	-15.924	38.289
11 - 1	-46.340	-9.562	27.216

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: OC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 50 MSE= 502.8752
Critical Value of Studentized Range= 4.682

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
5 - 9	-49.512	11.100	71.712
5 - 10	-40.539	13.673	67.886
5 - 7	-37.959	16.253	70.466
5 - 8	-44.178	16.433	77.045
5 - 3	-27.456	22.801	73.057
5 - 6	-36.878	23.733	84.345
5 - 1	-15.593	30.095	75.783
5 - 11	-14.555	39.657	93.870
5 - 2	-19.645	40.967	101.578
9 - 5	-71.712	-11.100	49.512
9 - 10	-51.639	2.573	56.786
9 - 7	-49.059	5.153	59.366
9 - 8	-55.278	5.333	65.945
9 - 3	-38.556	11.701	61.957
9 - 6	-47.978	12.633	73.245
9 - 1	-26.693	18.995	64.683
9 - 11	-25.655	28.557	82.770
9 - 2	-30.745	29.867	90.478
10 - 5	-67.886	-13.673	40.539
10 - 9	-56.786	-2.573	51.639
10 - 7	-44.370	2.580	49.530
10 - 8	-51.453	2.760	56.973
10 - 3	-33.192	9.127	51.447
10 - 6	-44.153	10.060	64.273
10 - 1	-20.356	16.422	53.200
10 - 11	-20.966	25.984	72.934
10 - 2	-26.919	27.293	81.506
7 - 5	-70.466	-16.253	37.959
7 - 9	-59.366	-5.153	49.059
7 - 10	-49.530	-2.580	44.370
7 - 8	-54.033	0.180	54.393
7 - 3	-35.772	6.547	48.867
7 - 6	-46.733	7.480	61.693
7 - 1	-22.936	13.842	50.620
7 - 11	-23.546	23.404	70.354
7 - 2	-29.499	24.713	78.926

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
11 - 2	-52.903	1.309	55.522	
2 - 5	-101.578	-40.967	19.645	
2 - 9	-90.478	-29.867	30.745	
2 - 10	-81.506	-27.293	26.919	
2 - 7	-78.926	-24.713	29.499	
2 - 8	-85.145	-24.533	36.078	
2 - 3	-68.422	-18.166	32.091	
2 - 6	-77.845	-17.233	43.378	
2 - 1	-56.559	-10.872	34.816	
2 - 11	-55.522	-1.309	52.903	

units: %
mandatory for sample A1, B1 and B2 (if pH > 6)
reported by 20 laboratories

TABLE I : CaCO₃ - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1	A1	3	N/R
2	A1	3	R
3			
4	A1	2	N
5	A1	1 not reported	N
6	A1	2	N/R
7	A1	3	N=R
8	A1	3	N/R
9	A1	3	N=R
10	A1	3	N
11	A1	3	R
12	A1	3	R
13	A1	3	R
14			
15	A1	3	R
16	A1	2	N=R
17	A1	3	N
18	A1 A2 B1 B2	1 not reported	R
19	A1	3	R
20	A1	1 not reported	R
21	A1	3	N
22	A1	3	R

TABLE II : CaCO₃ - Summary method description

method	description
1	reference
2	wet ashing
3	gravimetric determination
4	T.N.V. method
5	soil + H ₂ O + KOH + HCL + BaCl ₂
6	titration HCl ratio 1:3
7	CARMHOMAT 8-AD6
8	unspecified

TABLE IIIa : CaCO3 - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	26.00	26.47	28.90	0.45	1.70
2							
3							
4	3	2	26.44	26.75	27.05	0.43	1.61
5	8	1	23.30	23.30			
6	4	2	26.30	26.50	26.70	0.28	1.07
7							
8	8	3	14.50	14.77	15.00	0.25	1.70
9							
10							
11	7	3	23.30	25.00	26.70	1.70	6.80
12							
13							
14							
15							
16							
17	5	3	6.00	6.03	6.10	0.06	0.96
18							
19							
20							
21	6	3	17.80	17.80	17.80	0.00	0.00
22							

TABLE IIIb : CaCO3 - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1	3	26.00	26.17	26.50	0.29	1.10
2	2	8.10	8.10	8.10		
3						
4						
5						
6	2	26.80	27.10	27.40	0.42	1.57
7	3	24.20	24.57	25.00	0.40	1.65
8	3	14.50	14.77	15.00	0.25	1.70
9	3	25.50	25.77	26.10	0.31	1.19
10	3	9.00	9.00	9.00	0.00	0.00
11						
12	3	24.70	27.70	29.90	2.69	9.71
13	3	26.60	27.17	27.50	0.49	1.82
14						
15	3	9.69	10.16	10.70	0.51	5.00
16	2	27.20	27.30	27.40	0.14	0.52
17						
18	1		25.00			
19	3		27.60			
20	1		21.00			
21						
22	3	17.40	17.40	17.40	0.00	0.00

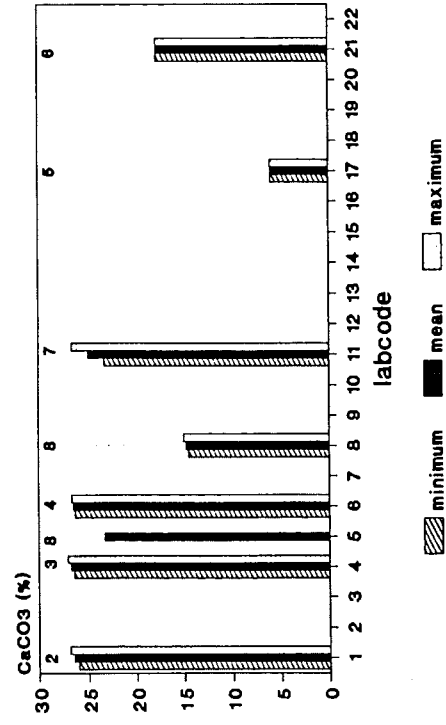
sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
15	2	0.49	0.50	0.50	0.01	1.43
18	1		0.10			

sample B1 & B2 - reference method

labcode	sample	repl	minimum	mean	maximum	std	cv
18	B1	1		0.10			
18	B2	1		0.10			

CaCO₃ sample A1
national methods



CaCO₃ sample A1
reference method

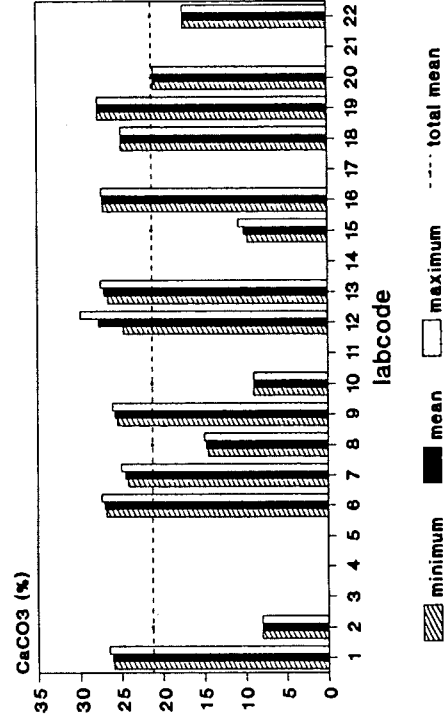


TABLE V : CaCO3 Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 2 3 4 5 6 7 8

Number of observations in by group = 58

Dependent Variable: CaCO3

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	1036.34937120	4.05	0.0014
Error	50	1825.66127018		
Corrected Total	57	2862.01064138		
R-Square				
			C.V.	CaCO3 Mean
			0.362105	20.85310345

Source	DF	Type I SS	F Value	Pr > F
METH	7	1036.34937120	4.05	0.0014
Source	DF	Type III SS	F Value	Pr > F
METH	7	1036.34937120	4.05	0.0014

TABLE IV : CaCO3 - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	38	21.30	6.91	0.80	0.00
A1	2	3	26.47	0.45	1.00	0.88
A1	3	2	26.75	0.43	1.00	1.00
A1	4	2	26.50	0.28	1.00	1.00
A1	5	3	6.03	0.06	0.75	0.00
A1	6	3	17.80	0.00	0.00	0.00
A1	7	3	25.00	1.70	1.00	0.99
A1	8	4	16.90	4.27	0.68	0.01
A2	1	3	0.36	0.23	0.77	0.04
B1	1	1	0.10			
B2	1	1	0.10			

Remark:
Labcode 19 reported mean values for 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for method 1 are different from the real number of replicates.

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
6 - 3	-26.391	-8.945	-8.945	-26.391	8.501
6 - 4	-26.146	-8.700	-8.700	-26.146	8.746
6 - 2	-24.271	-8.667	-8.667	-24.271	6.937
6 - 7	-22.804	-7.200	-7.200	-22.804	8.404
6 - 1	-14.963	-3.502	-3.502	-14.963	7.959
6 - 8	-13.696	0.900	0.900	-13.696	15.496
6 - 5	-3.837	11.767	11.767	-3.837	27.371
8 - 3	-26.395	-9.845	-9.845	-26.395	6.705
8 - 4	-26.150	-9.600	-9.600	-26.150	6.950
8 - 2	-24.163	-9.567	-9.567	-24.163	5.029
8 - 7	-22.696	-8.100	-8.100	-22.696	6.496
8 - 1	-14.448	-4.402	-4.402	-14.448	5.643
8 - 6	-15.496	-0.900	-0.900	-15.496	13.696
8 - 5	-3.729	10.867	10.867	-3.729	25.463
5 - 3	-38.157	-20.712	-20.712	-38.157	-3.266
5 - 4	-37.912	-20.467	-20.467	-37.912	-3.021
5 - 2	-36.037	-20.433	-20.433	-36.037	-4.829
5 - 7	-34.571	-18.967	-18.967	-34.571	-3.363
5 - 1	-26.730	-15.269	-15.269	-26.730	-3.808
5 - 6	-27.371	-11.767	-11.767	-27.371	3.837
5 - 8	-25.463	-10.867	-10.867	-25.463	3.729

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CaCO₃

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 50 MSE= 36.51323
Critical Value of Studentized Range= 4.473

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
3 - 4	-18.866	0.245	0.245	19.356	
3 - 2	-17.167	0.278	0.278	17.724	
3 - 7	-15.701	1.745	1.745	19.191	
3 - 1	-8.422	5.443	5.443	19.307	
3 - 6	-8.501	8.945	8.945	26.391	
3 - 8	-6.705	9.845	9.845	26.395	
3 - 5	3.266	20.712	20.712	38.157	***
4 - 3	-19.356	-0.245	-0.245	18.866	
4 - 2	-17.412	0.033	0.033	17.479	
4 - 7	-15.946	1.500	1.500	18.946	
4 - 1	-8.667	5.198	5.198	19.062	
4 - 6	-8.746	8.700	8.700	26.146	
4 - 8	-6.950	9.600	9.600	26.150	
4 - 5	3.021	20.467	20.467	37.912	***
2 - 3	-17.724	-0.278	-0.278	17.167	
2 - 4	-17.479	-0.033	-0.033	17.412	
2 - 7	-14.137	1.467	1.467	17.071	
2 - 1	-6.297	5.164	5.164	16.625	
2 - 6	-6.937	8.667	8.667	24.271	
2 - 8	-5.029	9.567	9.567	24.163	
2 - 5	4.829	20.433	20.433	36.037	***
7 - 3	-19.191	-1.745	-1.745	15.701	
7 - 4	-18.946	-1.500	-1.500	15.946	
7 - 2	-17.071	-1.467	-1.467	14.137	
7 - 1	-7.763	3.698	3.698	15.159	
7 - 6	-8.404	7.200	7.200	22.804	
7 - 8	-6.496	8.100	8.100	22.696	
7 - 5	3.363	18.967	18.967	34.571	***
1 - 3	-19.307	-5.443	-5.443	8.422	
1 - 4	-19.062	-5.198	-5.198	8.667	
1 - 2	-16.625	-5.164	-5.164	6.297	
1 - 7	-15.159	-3.698	-3.698	7.763	
1 - 6	-7.959	3.502	3.502	14.963	
1 - 8	-5.643	4.402	4.402	14.448	
1 - 5	3.808	15.269	15.269	26.730	***

5. ELECTRICAL CONDUCTIVITY

units: mS/m
optional for all samples
reported by 5 laboratories

TABLE I : Electrical conductivity - Overview of reported samples

labcode	samples reported	number of replicates
1		
2		
3		
4		
5		
6	A1 A2 B1 B2	2
7	A1 A2 B1 B2	3
8		
9	A1 A2 B1 B2	3
10		
11		
12		
13		
14		
15	A1 A2 B1 B2	3 A2: 2
16		
17		
18	A1 A2 B1 B2	1 not reported
19		
20		
21		
22		

TABLE II : Electrical conductivity - Summary method description

method	description
1	20 g sample - 100 g DI-water
2	AFNOR X31-113
3	10 g sample - 100 ml
4	20 g sample - 100 g DI-water, separated from soil

TABLE III : Electrical conductivity - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6	1	2	23.00	23.50	24.00	0.71	3.01
7	2	3	18.30	18.50	18.80	0.27	1.43
8							
9	3	3	12.00	12.07	12.10	0.06	0.48
10							
11							
12							
13							
14							
15	1	3	18.60	18.63	20.50	0.78	3.97
16							
17							
18	1	1		16.00			
18	4	1		21.00			
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6	1	2	28.00	28.40	28.80	0.57	1.99
7	2	3	21.20	21.83	22.40	0.60	2.76
8							
9							
10							
11							
12							
13							
14							
15	1	2	82.00	82.25	82.50	0.25	0.30
16							
17							
18	1	1		60.50			
18	4	1		98.60			
19							
20							
21							
22							

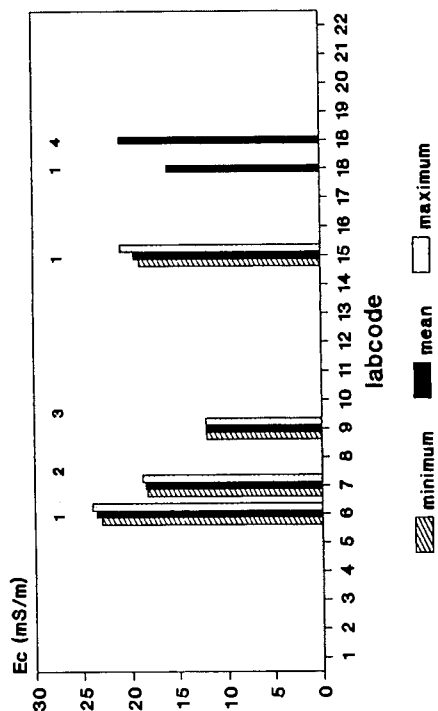
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6	1	2	5.31	5.42	5.53	0.16	2.87
7	2	3	5.50	5.53	5.60	0.06	1.04
8							
9	3	3	3.50	3.60	3.70	0.10	2.78
10							
11							
12							
13							
14							
15	1	3	5.80	6.37	7.30	0.66	10.36
16							
17							
18	1	1		4.60			
18	4	1		4.90			
19							
20							
21							
22							

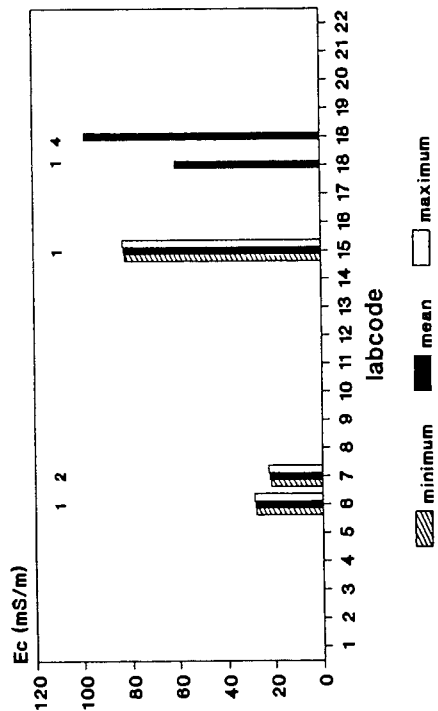
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6	1	2	10.47	10.65	10.83	0.26	2.39
7	2	3	11.80	11.87	12.00	0.12	0.97
8							
9	3	3	4.50	4.80	5.00	0.27	5.51
10							
11							
12							
13							
14							
15	1	3	11.30	11.90	12.80	0.65	5.46
16							
17							
18	1	1		9.30			
18	4	1		9.50			
19							
20							
21							
22							

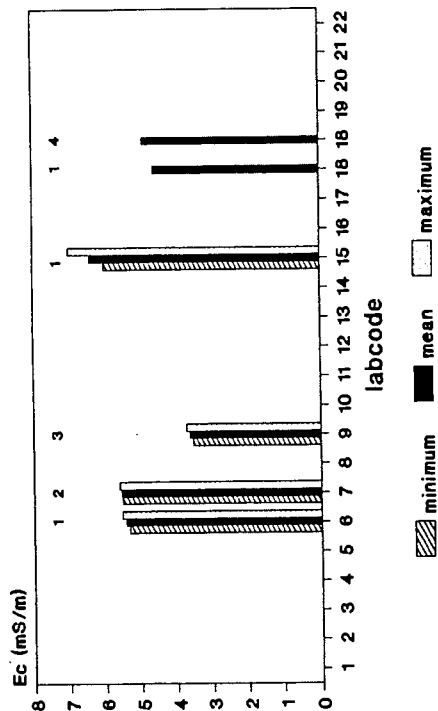
Ec sample A1
national methods



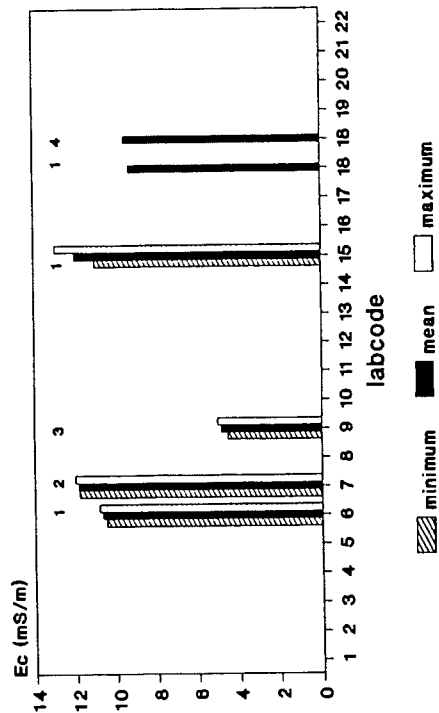
Ec sample A2
national methods



Ec sample B1
national methods



Ec sample B2
national methods



----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	4	1 2 3 4

Number of observations in by group = 13

Dependent Variable: EC

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	152.64102564	11.00	0.0023
Error	9	41.64666667		

Corrected Total

12

194.28769231

R-Square

C.V.

EC Mean

0.785644

11.86459

18.13076923

Source

DF

Type I SS

F Value

Pr > F

METH

3

152.64102564

11.00

0.0023

Source

DF

Type III SS

F Value

Pr > F

METH

3

152.64102564

11.00

0.0023

TABLE IV : Electrical conductivity - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	6	20.50	2.88	0.97	0.91
A1	2	3	18.50	0.27	0.89	0.36
A1	3	3	12.07	0.06	0.75	0.00
A1	4	1	21.00			
A2	1	5	56.46	27.15	0.83	0.13
A2	2	3	21.83	0.60	0.99	0.82
A2	4	1	98.60			
B1	1	6	5.74	0.81	0.97	0.90
B1	2	3	5.53	0.06	0.75	0.00
B1	3	3	3.60	0.10	1.00	0.99
B1	4	1	4.90			
B2	1	6	10.37	2.83	0.80	0.05
B2	2	3	11.87	0.12	0.75	0.00
B2	3	3	4.80	0.27	0.89	0.36
B2	4	1	9.50			

----- SAMPLE=A2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	4	1 2 3 4

Number of observations in by group = 10

NOTE: Due to missing values, only 9 observations can be used in this analysis.

Dependent Variable: EC

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	4949.26133333	5.04	0.0520
Error	6	2948.75866667		
Corrected Total	8	7898.02000000		

R-Square	C.V.	EC Mean
0.626646	44.69535	49.60000000

Source	DF	Type I SS	F Value	Pr > F
METH	2	4949.26133333	5.04	0.0520

Source	DF	Type III SS	F Value	Pr > F
METH	2	4949.26133333	5.04	0.0520

----- SAMPLE=A1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 9 MSE= 4.627407
Critical Value of Studentized Range= 4.415

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Confidence Limit		Difference Between Means	Simultaneous Upper Confidence Limit		
	Lower	Upper		Lower	Upper	
4 - 1	-6.754	0.500	0.500	7.754		
4 - 2	-5.254	2.500	2.500	10.254		
4 - 3	1.179	8.933	8.933	16.688		***
1 - 4	-7.754	-0.500	-0.500	6.754		
1 - 2	-2.749	2.000	2.000	6.749		
1 - 3	3.685	8.433	8.433	13.182		***
2 - 4	-10.254	-2.500	-2.500	5.254		
2 - 1	-6.749	-2.000	-2.000	2.749		
2 - 3	0.950	6.433	6.433	11.916		***
3 - 4	-16.688	-8.933	-8.933	-1.179		***
3 - 1	-13.182	-8.433	-8.433	-3.685		***
3 - 2	-11.916	-6.433	-6.433	-0.950		***

----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	4	1 2 3 4

Number of observations in by group = 13

Dependent Variable: EC				
Source	DF	Sum of Squares	F Value	Pr > F
Model	3	9.79604103	8.97	0.0046
Error	9	3.27806667		
Corrected Total	12	13.07410769		
R-Square C.V. EC Mean				
		0.749270	11.75560	5.13384615

Source	DF	Type I SS	F Value	Pr > F
METH	3	9.79604103	8.97	0.0046
Source	DF	Type III SS	F Value	Pr > F
METH	3	9.79604103	8.97	0.0046

----- SAMPLE=A2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 6 MSE= 491.4598
Critical Value of Studentized Range= 4.339

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means		Lower Confidence Limit	Upper Confidence Limit
4 - 1	-32.38	42.14	76.77		116.66	
4 - 2	-1.78	76.77			155.31	
1 - 4	-116.66	-42.14			32.38	
1 - 2	-15.05	34.63			84.30	
2 - 4	-155.31	-76.77			1.78	
2 - 1	-84.30	-34.63			15.05	

----- SAMPLE=B1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 9 MSE= 0.36423
Critical Value of Studentized Range= 4.415

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
1	- 2	-1.126		0.207		1.539	
1	- 4	-1.195		0.840		2.875	
1	- 3	0.808		2.140	***	3.472	***
2	- 1	-1.539		-0.207		1.126	
2	- 4	-1.542		0.633		2.809	
2	- 3	0.395		1.933	***	3.472	***
4	- 1	-2.875		-0.840		1.195	
4	- 2	-2.809		-0.633		1.542	
4	- 3	-0.876		1.300		3.476	
3	- 1	-3.472		-2.140		-0.808	***
3	- 2	-3.472		-1.933		-0.395	***
3	- 4	-3.476		-1.300		0.876	

----- SAMPLE=B2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	4	1 2 3 4

Number of observations in by group = 13

Dependent Variable: EC						
Source	DF	Sum of Squares	F Value	Pr > F		
Model	3	87.33076923	6.50	0.0124		
Error	9	40.27980000				
Corrected Total	12	127.61056923				
				R-Square	C.V.	EC Mean
				0.684354	22.59827	9.36153846
Source	DF	Type I SS	F Value	Pr > F		
METH	3	87.33076923	6.50	0.0124		
Source	DF	Type III SS	F Value	Pr > F		
METH	3	87.33076923	6.50	0.0124		

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 9 MSE= 4.475533
Critical Value of Studentized Range= 4.415

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Simultaneous Upper Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
2 - 1	-3.170	6.170	1.500	6.170		
2 - 4	-5.259	9.993	2.367	9.993		
2 - 3	1.674	12.459	7.067	12.459		***
1 - 2	-6.170	3.170	-1.500	3.170		
1 - 4	-6.267	8.000	0.867	8.000		
1 - 3	0.897	10.237	5.567	10.237		***
4 - 2	-9.993	5.259	-2.367	5.259		
4 - 1	-8.000	6.267	-0.867	6.267		
4 - 3	-2.926	12.326	4.700	12.326		
3 - 2	-12.459	-1.674	-7.067	-1.674		***
3 - 1	-10.237	-0.897	-5.567	-0.897		***
3 - 4	-12.326	2.926	-4.700	2.926		

6. Particle size distribution CLAY

units: %
reported by 12 laboratories

TABLE I : Particle size distribution clay - Overview of reported data

labcode	samples reported	number of replicates
1	A1 B1 B2	3
2	A1 B1 B2	3
3		
4		
5	B1 B2	B1: 2 B2: 1
6		
7	A1 B1 B2	3
8	A1 B1 B2	1
9	A1 B1 B2	3
10	A1 B1 B2	3
11		
12	A1 B1 B2	3
13		
14	A1 B1 B2	3
15	A1 B1 B2	2
16		
17		
18		
19	A1 B1 B2	3
20		
21	A1 B1 B2	3
22		

TABLE II : Particle size distribution clay - Summary method descriptions

method	description
1	reference, ISRIC 1987
2	hydrometer, without pre-treatment
3	AFNOR X31-107
4	areometer
5	determination without destruction of carbonates and organic matter
6	unspecified

TABLE III : Particle size distribution clay - Descriptive statistics

sample A1 - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	6	3	24.00	24.27	24.80	0.46	1.90
2	2	3	19.30	20.70	22.40	1.57	7.59
3							
4							
5							
6							
7	3	3	22.80	23.00	23.20	0.20	0.87
8	6	1		28.00			
9	5	3	10.00	10.87	11.00	0.58	5.41
10	1	3	14.00	15.23	16.00	1.08	7.08
11							
12	1	3	17.40	18.03	19.00	0.85	4.72
13							
14	4	3	18.30	22.17	26.20	5.20	23.45
15	1	2	22.30	22.40	22.50	0.14	0.63
16							
17							
18							
19	1	3		9.70			
20							
21	1	3	13.90	15.27	16.00	1.19	7.76
22							

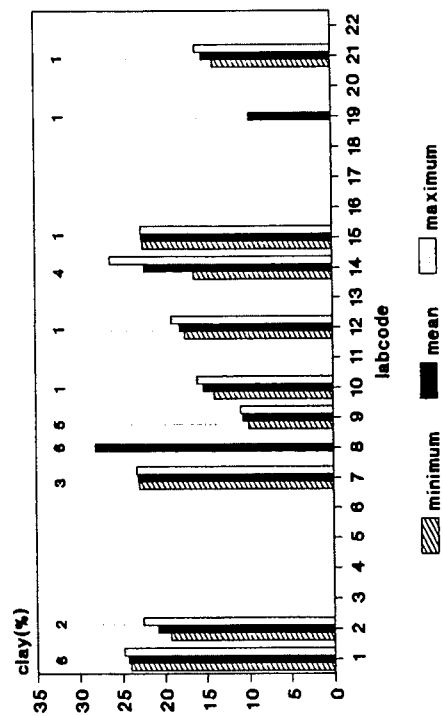
sample B1 - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	6	3	25.90	26.50	27.40	0.79	3.00
2	2	3	19.30	19.97	20.30	0.58	2.89
3							
4							
5	6	2		23.40			
6							
7	3	3	22.80	23.23	23.90	0.59	2.52
8	6	1		27.00			
9	5	3	22.00	22.00	22.00	0.00	0.00
10	1	3	21.60	21.83	22.10	0.25	1.15
11							
12	1	3	21.20	21.47	21.90	0.38	1.76
13							
14	4	3	22.70	22.97	23.20	0.25	1.10
15	1	2	24.96	25.01	25.05	0.06	0.26
16							
17							
18							
19	1	3		20.30			
20							
21	1	3	15.30	16.93	19.00	1.89	11.15
22							

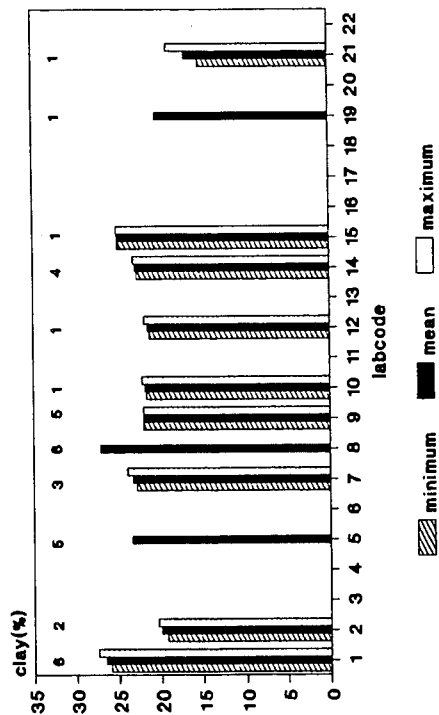
sample B2 - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	6	3	15.30	16.70	17.60	1.23	7.36
2	2	2	1.00	1.50	2.00	0.71	47.14
3							
4							
5	6	1		2.10			
6							
7	3	3	8.20	8.33	8.50	0.15	1.83
8	6	1		5.10			
9	5	3	13.00	14.00	15.00	1.00	7.14
10	1	3	7.40	8.50	9.20	0.98	11.35
11							
12	1	2	0.80	1.10	1.40	0.42	38.57
13							
14	4	3	3.50	3.57	3.60	0.06	1.62
15	1	2	5.09	5.36	5.62	0.38	7.00
16							
17							
18							
19	1	3		12.50			
20							
21	1	3	0.10	0.17	0.20	0.06	34.64
22							

clay sample A1
fraction <2μm



clay sample B1
fraction <2μm



clay sample B2
fraction <2μm

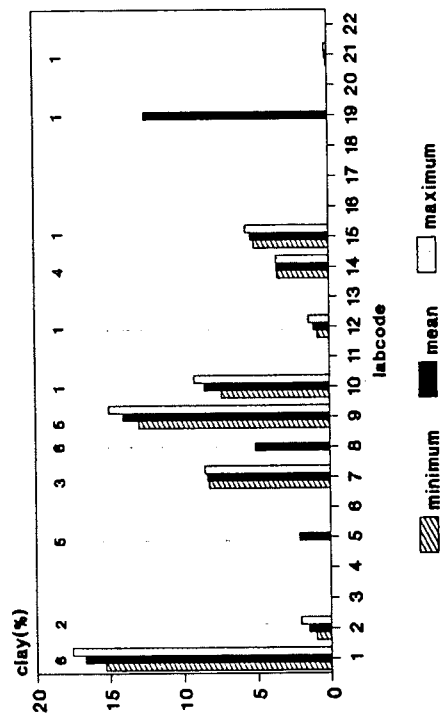


TABLE V : Clay Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 6

Number of observations in by group = 28

Dependent Variable: CLAY

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	500.43380952	8.73	0.0001
Error	22	252.33583333		
Corrected Total	27	752.76964286		

R-Square	C.V.	CLAY Mean
0.664790	17.77468	19.05357143

Source	DF	Type I SS	F Value	Pr > F
METH	5	500.43380952	8.73	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	5	500.43380952	8.73	0.0001

TABLE IV : Particle size distribution clay - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	12	16.93	4.06	0.94	0.43
A1	2	3	20.70	1.57	0.97	0.68
A1	3	3	23.00	0.20	1.00	0.99
A1	4	3	22.17	5.20	0.91	0.41
A1	5	3	10.67	0.58	0.75	0.00
A1	6	4	25.20	1.90	0.77	0.05
B1	1	12	20.92	2.90	0.92	0.24
B1	2	3	19.97	0.58	0.75	0.00
B1	3	3	23.23	0.59	0.88	0.33
B1	4	3	22.97	0.25	0.99	0.78
B1	5	3	22.00	0.00		
B1	6	5	25.98	1.56	0.88	0.29
B2	1	11	4.67	4.41	0.89	0.13
B2	2	2	1.50	0.71	1.00	1.00
B2	3	3	8.33	0.15	0.96	0.64
B2	4	3	3.57	0.06	0.75	0.00
B2	5	3	14.00	1.00	1.00	0.99
B2	6	5	11.46	7.30	0.82	0.11

Remark:

Labcode 19 reported mean values for 3 numbers of replicates.

To calculate the total number of replicates for the method-grouped laboratories,

SAS counts the number of reported results.

So, the "repl" values in this table for method 1 are different from the real number of replicates.

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class Levels Values
METH 6 1 2 3 4 5 6

Number of observations in by group = 29

Dependent Variable: CLAY					
Source	DF	Sum of Squares	F Value	Pr > F	
Model	5	111.14029224	4.93	0.0033	
Error	23	103.63042500			
Corrected Total	28	214.77071724			
R-Square C.V. CLAY Mean					
		9.537664	22.25551724		
Type I SS F Value Pr > F					
Source	DF	Type I SS	F Value	Pr > F	
METH	5	111.14029224	4.93	0.0033	
Type III SS F Value Pr > F					
Source	DF	Type III SS	F Value	Pr > F	
METH	5	111.14029224	4.93	0.0033	

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CLAY

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 22 MSE= 11.46981
Critical Value of Studentized Range= 4.405

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
6 - 3	-5.858	2.200	10.258	5.858	-2.200	5.858
6 - 4	-5.024	3.033	11.091	9.447	0.833	7.781
6 - 2	-3.558	4.500	12.558	10.914	2.300	6.314
6 - 1	2.184	8.275	14.366	12.885	6.075	12.885
6 - 5	6.476	14.533	22.591	20.947	12.333	20.947
3 - 6	-10.258	-2.200	5.858	5.024	-3.033	-0.833
3 - 4	-7.781	0.833	9.447	7.781	-0.833	7.781
3 - 2	-6.314	2.300	10.914	10.081	1.467	10.081
3 - 1	-0.735	6.075	12.885	12.052	5.242	12.052
3 - 5	3.719	12.333	20.947	20.114	11.500	20.114
4 - 6	-11.091	-3.033	5.024	3.558	-4.500	3.558
4 - 3	-9.447	-0.833	7.781	6.314	-2.300	6.314
4 - 2	-7.147	1.467	10.081	7.147	-1.467	7.147
4 - 1	-1.568	5.242	12.052	10.585	3.775	10.585
4 - 5	2.886	11.500	20.114	18.647	10.033	18.647
2 - 6	-12.558	-4.500	3.558	-2.184	-8.275	2.184
2 - 3	-10.914	-2.300	6.314	0.735	-6.075	0.735
2 - 4	-10.081	-1.467	7.147	1.568	-5.242	1.568
2 - 1	-3.035	3.775	10.585	3.035	-3.775	3.035
2 - 5	1.419	10.033	18.647	13.068	6.258	13.068
1 - 6	-14.366	-8.275	-2.184	-6.476	-14.533	-6.476
1 - 3	-12.885	-6.075	0.735	-3.719	-12.333	-3.719
1 - 4	-12.052	-5.242	1.568	-2.886	-11.500	-2.886
1 - 2	-10.585	-3.775	3.035	-1.419	-10.033	-1.419
1 - 5	-0.552	6.258	13.068	0.552	-6.258	0.552
5 - 6	-22.591	-14.533	-6.476	***	***	***
5 - 3	-20.947	-12.333	-3.719	***	***	***
5 - 4	-20.114	-11.500	-2.886	***	***	***
5 - 2	-18.647	-10.033	-1.419	***	***	***
5 - 1	-13.068	-6.258	0.552	***	***	***

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 6

Number of observations in by group = 28

NOTE: Due to missing values, only 27 observations can be used in this analysis.

Dependent Variable: CLAY

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	407.18008620	4.16	0.0087
Error	21	410.77618788		
Corrected Total	26	817.95627407		

R-Square	C.V.	CLAY Mean
0.497802	63.04546	7.01518519

Source	DF	Type I SS	F Value	Pr > F
METH	5	407.18008620	4.16	0.0087

Source	DF	Type III SS	F Value	Pr > F
METH	5	407.18008620	4.16	0.0087

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CLAY

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 23 MSE= 4.505671
Critical Value of Studentized Range= 4.388

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Difference	Lower Confidence Limit	Upper Confidence Limit
6 - 3	-2.064	2.747	2.747	7.557	7.557	7.824
6 - 4	-1.797	3.013	3.013	7.824	7.824	8.790
6 - 5	-0.830	3.980	3.980	8.790	8.790	***
6 - 1	1.557	5.062	5.062	8.568	8.568	***
6 - 2	1.203	6.013	6.013	10.824	10.824	***
3 - 6	-7.557	-2.747	-2.747	2.064	2.064	
3 - 4	-5.111	0.267	0.267	5.645	5.645	
3 - 5	-4.145	1.233	1.233	6.611	6.611	
3 - 1	-1.936	2.316	2.316	6.567	6.567	
3 - 2	-2.111	3.267	3.267	8.645	8.645	
4 - 6	-7.824	-3.013	-3.013	1.797	1.797	
4 - 3	-5.645	-0.267	-0.267	5.111	5.111	
4 - 5	-4.411	0.967	0.967	6.345	6.345	
4 - 1	-2.202	2.049	2.049	6.301	6.301	
4 - 2	-2.378	3.000	3.000	8.378	8.378	
5 - 6	-8.790	-3.980	-3.980	0.830	0.830	
5 - 3	-6.611	-1.233	-1.233	4.145	4.145	
5 - 4	-6.345	-0.967	-0.967	4.411	4.411	
5 - 1	-3.169	1.082	1.082	5.334	5.334	
5 - 2	-3.345	2.033	2.033	7.411	7.411	
1 - 6	-8.568	-5.062	-5.062	-1.557	-1.557	***
1 - 3	-6.567	-2.316	-2.316	1.936	1.936	
1 - 4	-6.301	-2.049	-2.049	2.202	2.202	
1 - 5	-5.334	-1.082	-1.082	3.169	3.169	
1 - 2	-3.301	0.951	0.951	5.202	5.202	
2 - 6	-10.824	-6.013	-6.013	-1.203	-1.203	***
2 - 3	-8.645	-3.267	-3.267	2.111	2.111	
2 - 4	-8.378	-3.000	-3.000	2.378	2.378	
2 - 5	-7.411	-2.033	-2.033	3.345	3.345	
2 - 1	-5.202	-0.951	-0.951	3.301	3.301	

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CLAY

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 21 MSE= 19.56077
Critical Value of Studentized Range= 4.424

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Upper Confidence Limit
5 - 6	-7.565	2.540	2.540	12.645	
5 - 3	-5.631	5.667	5.667	16.964	
5 - 1	0.314	9.326	9.326	18.339	***
5 - 4	-0.864	10.433	10.433	21.731	
5 - 2	-0.131	12.500	12.500	25.131	
6 - 5	-12.645	-2.540	-2.540	7.565	
6 - 3	-6.978	3.127	3.127	13.231	
6 - 1	-0.677	6.786	6.786	14.249	
6 - 4	-2.211	7.893	7.893	17.998	
6 - 2	-1.616	9.960	9.960	21.536	
3 - 5	-16.964	-5.667	-5.667	5.631	
3 - 6	-13.231	-3.127	-3.127	6.978	
3 - 1	-5.353	3.660	3.660	12.672	
3 - 4	-6.531	4.767	4.767	16.064	
3 - 2	-5.798	6.833	6.833	19.464	
1 - 5	-18.339	-9.326	-9.326	-0.314	***
1 - 6	-14.249	-6.786	-6.786	0.677	
1 - 3	-12.672	-3.660	-3.660	5.353	
1 - 4	-7.905	1.107	1.107	10.119	
1 - 2	-7.463	3.174	3.174	13.810	
4 - 5	-21.731	-10.433	-10.433	0.864	
4 - 6	-17.998	-7.893	-7.893	2.211	
4 - 3	-16.064	-4.767	-4.767	6.531	
4 - 1	-10.119	-1.107	-1.107	7.905	
4 - 2	-10.564	2.067	2.067	14.698	
2 - 5	-25.131	-12.500	-12.500	0.131	
2 - 6	-21.536	-9.960	-9.960	1.616	
2 - 3	-19.464	-6.833	-6.833	5.798	
2 - 1	-13.810	-3.174	-3.174	7.463	
2 - 4	-14.698	-2.067	-2.067	10.564	

7. Particle size distribution SILT

units: %
reported by 11 laboratories

TABLE I : Particle size distribution SILT - Overview of reported data

labcode	samples reported	number of replicates	fraction
1	A1 B1 B2	3	2-50µm
2			
3			
4	B1 B2	B1:2 B2: 1	2-63µm
5			
6			
7	A1 B1 B2	3	2-50µm
8	A1 B1 B2	1	
9	A1 B1 B2	3	2-63µm
10	A1 B1 B2	3	2-63µm
11			
12	A1 B1 B2	3	2-63µm
13			
14	A1 B1 B2	3	2-50µm
15	A1 B1 B2	2	2-50µm
16			
17			
18			
19	A1 B1 B2	3	2-63µm
20			
21	A1 B1 B2	3	2-63µm
22			

TABLE II : Particle size distribution silt - Summary method descriptions

method	description
1	reference, ISRIC 1987
2	hydrometer, without pre-treatment
3	AFNOR X31-107
4	areometer
5	determination without destruction of carbonates and organic matter
6	unspecified

TABLE III : Particle size distribution silt - Descriptive statistics

sample A1 - fraction 2-50µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	59.00	60.40	62.10	1.57	2.60
3							
4							
5							
6							
7	3	3	61.80	62.07	62.30	0.25	0.41
8							
9							
10							
11							
12							
13							
14	4	3	55.00	58.70	63.30	4.22	7.19
15	1	2	52.63	52.63	52.63	0.00	0.00
16							
17							
18							
19							
20							
21							
22							

sample B1 - fraction 2-50µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	70.20	70.87	71.20	0.58	0.82
3							
4							
5							
6							
7	3	3	71.40	71.90	72.70	0.70	0.97
8							
9							
10							
11							
12							
13							
14	4	3	75.10	75.27	75.50	0.21	0.26
15	1	2	64.81	65.50	66.19	0.98	1.49
16							
17							
18							
19							
20							
21							
22							

sample B2 - fraction 2-50µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	2	13.20	13.70	14.20	0.71	5.16
3							
4							
5							
6							
7	3	3	5.50	6.83	7.90	1.22	17.86
8							
9							
10							
11							
12							
13							
14	4	3	14.00	14.23	14.60	0.32	2.26
15	1	2	6.63	7.61	8.59	1.39	18.21
16							
17							
18							
19							
20							
21							
22							

TABLE III : Particle size distribution silt - Descriptive statistics

sample A1 - fraction 2-63µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6							
7	3						
8	5	1		53.00			
9	5	3	72.00	73.00	73.00	0.58	0.80
10	1	3	66.70	67.83	68.90	1.10	1.62
11							
12	1	3	59.20	60.37	61.20	1.04	1.72
13							
14							
15							
16							
17							
18							
19	5	3		72.50			
20							
21	5	3	68.90	69.67	70.50	0.80	1.15
22							

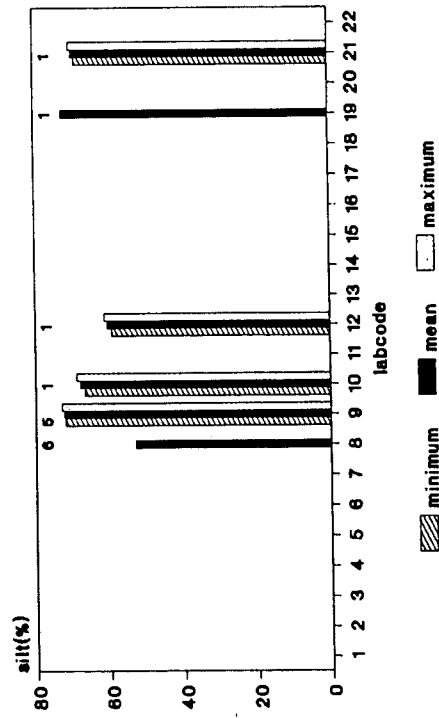
sample B1 - fraction 2-63µm - national/reference methods

labcode	meth	repl	min	mean	max	std	cv
1							
2							
3							
4							
5	5	1		72.60			
6							
7	3						
8	5	1		70.60			
9	5	3	76.00	76.00	76.00	0.00	0.00
10	1	3	72.00	73.53	74.90	1.46	1.98
11							
12	1	3	24.80	25.27	26.10	0.72	2.86
13							
14							
15							
16							
17							
18							
19	5	3		72.30			
20							
21	5	3	79.10	81.03	82.80	1.86	2.29
22							

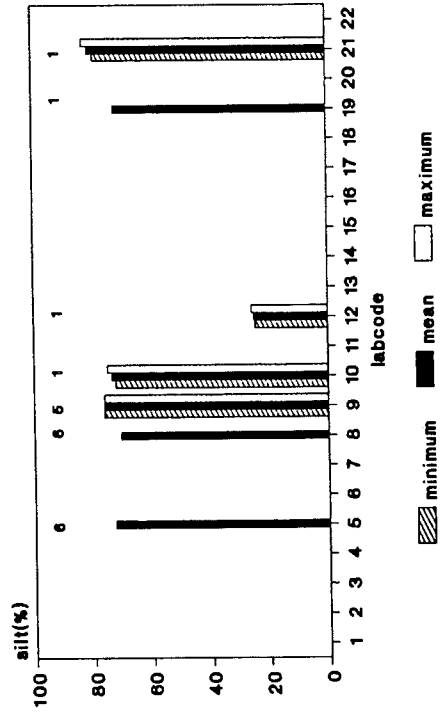
sample B2 - fraction 2-63µm - national/reference methods

labcode	meth	repl	min	mean	max	std	cv
1							
2							
3							
4							
5	5	1		16.40			
6							
7							
8	5	1		11.10			
9	5	3	10.00	10.00	10.00	0.00	0.00
10	1	3	12.10	12.23	12.30	0.12	0.94
11							
12	1	3	2.20	2.67	2.90	0.40	15.16
13							
14							
15							
16							
17							
18							
19	5	3		9.60			
20							
21	5	3	6.50	7.77	9.00	1.25	16.10
22							

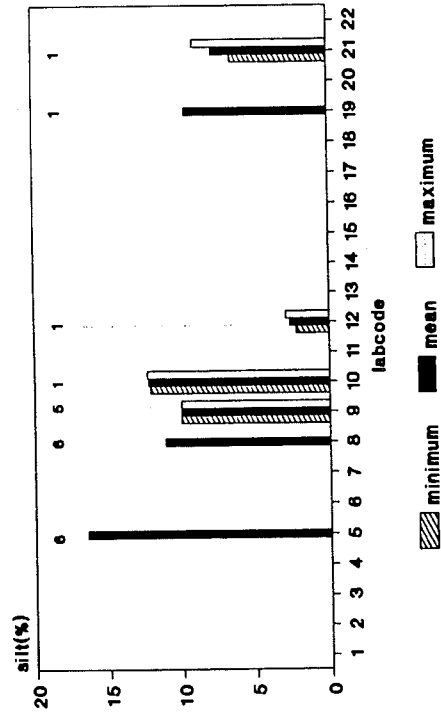
silt sample A1
fraction 2-63 μ



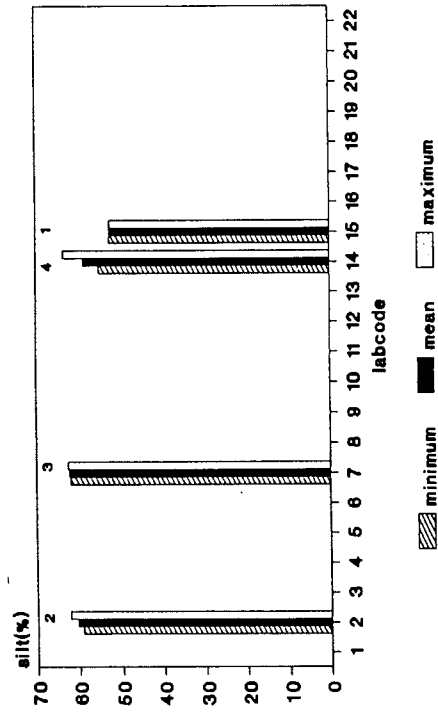
silt sample B1
fraction 2-63 μ



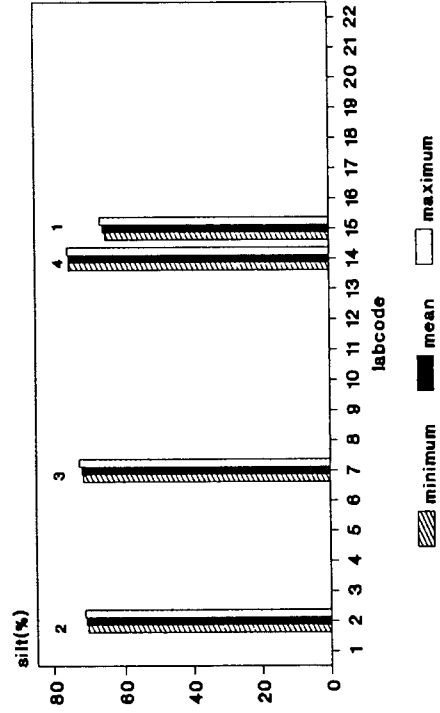
silt sample B2
fraction 2-63 μ



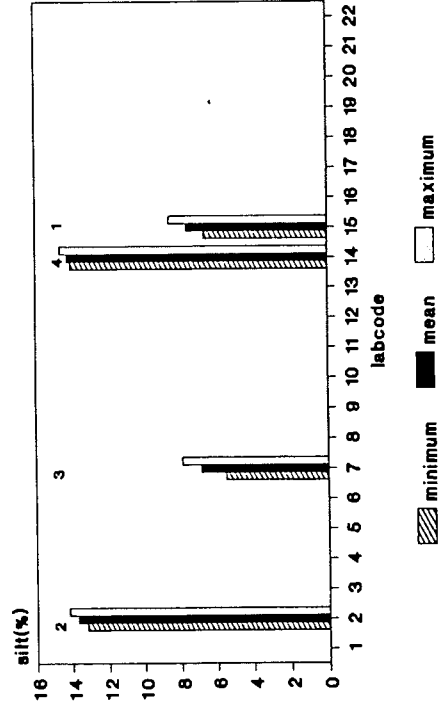
silt sample A1
fraction 2-50 μ



silt sample B1
fraction 2-50 μ



silt sample B2
fraction 2-50 μ



----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 25

NOTE: Due to missing values, only 14 observations can be used in this analysis.

Dependent Variable: SILT 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	282.59647619	8.15	0.0067
Error	11	190.69566667		
Corrected Total	13	473.29214286		

R-Square	C.V.	SILT 63 Mean
0.597087	6.227014	66.86428571

Source	DF	Type I SS	F Value	Pr > F
METHOD	2	282.59647619	8.15	0.0067
Source	DF	Type III SS	F Value	Pr > F
METHOD	2	282.59647619	8.15	0.0067

TABLE IV : Particle size distribution Silt - Total mean and normal distribution

fraction 2-63µm

sample	method	repl	mean	std	normal	prob<W
A1	1	10	66.61	4.60	0.89	0.15
A1	5	3	72.33	0.58	0.75	0.00
A1	6	1	53.00			
B1	1	10	61.18	25.05	0.72	0.00
B1	5	3	76.00	0.00		
B1	6	2	71.60	1.41	1.00	1.00
B2	1	10	7.76	4.01	0.89	0.15
B2	5	3	10.00	0.00		
B2	6	2	13.75	3.75	1.00	1.00

fraction 2-50µm

sample	method	repl	mean	std	normal	prob<W
A1	1	2	52.63	0.00		
A1	2	3	60.40	1.57	0.97	0.68
A1	3	3	62.07	0.25	0.99	0.78
A1	4	3	58.70	4.22	0.97	0.65
B1	1	2	65.50	0.98	1.00	1.00
B1	2	3	70.87	0.58	0.75	0.00
B1	3	3	71.90	0.70	0.86	0.27
B1	4	3	75.27	0.21	0.92	0.46
B2	1	2	7.61	1.39	1.00	1.00
B2	2	2	13.70	0.71	1.00	1.00
B2	3	3	6.83	1.22	0.96	0.64
B2	4	3	14.23	0.32	0.87	0.30

Remark:
Labcode 19 reported mean values for 3 replicates.
To calculate to total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for method 1 are different from the real number
of replicates.

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 26

NOTE: Due to missing values, only 15 observations can be used in this analysis.

Dependent Variable: SILT 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	591.77733333	0.63	0.5500
Error	12	5647.41600000		
Corrected Total	14	6239.19333333		
R-Square		C.V.		SILT 63 Mean
		33.10335		65.53333333

Source	DF	Type I SS	F Value	Pr > F
METHOD	2	591.77733333	0.63	0.5500
Source	DF	Type III SS	F Value	Pr > F
METHOD	2	591.77733333	0.63	0.5500

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SILT 63

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 11 MSE= 17.33597
Critical Value of Studentized Range= 3.820

Comparisons significant at the 0.05 level are indicated by '***'.

METHOD Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
5 - 1	-1.679	5.723	13.126	***
5 - 6	6.348	19.333	32.318	
1 - 5	-13.126	-5.723	1.679	***
1 - 6	1.816	13.610	25.404	
6 - 5	-32.318	-19.333	-6.348	***
6 - 1	-25.404	-13.610	-1.816	***

----- SAMPLE=B1 -----

General Linear Models Procedure
Tukey's Studentized Range (HSD) Test for variable: SILT 63

NOTE: This test controls the type I experimentwise error rate.
Alpha= 0.05 Confidence= 0.95 df= 12 MSE= 470.618
Critical Value of Studentized Range= 3.773

Comparisons significant at the 0.05 level are indicated by '***'.

METHOD Comparison	Simultaneous		Difference		Simultaneous
	Lower Limit	Confidence Limit	Between Means	Upper Confidence Limit	
5 - 6	-48.434	-23.279	4.400	57.234	
5 - 1			14.820	52.919	
6 - 5	-57.234	-34.411	-4.400	48.434	
6 - 1			10.420	55.251	
1 - 5	-52.919	-14.820	-14.820	23.279	
1 - 6	-55.251	-10.420	-10.420	34.411	

----- SAMPLE=B2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 26

NOTE: Due to missing values, only 15 observations can be used in this analysis.

Dependent Variable: SILT 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	63.50033333	2.40	0.1328
Error	12	158.76900000		
Corrected Total	14	222.26933333		

R-Square	C.V.	SILT 63 Mean
0.285691	40.38575	9.00666667

Source	DF	Type I SS	F Value	Pr > F
METHOD	2	63.50033333	2.40	0.1328
Source	DF	Type III SS	F Value	Pr > F
METHOD	2	63.50033333	2.40	0.1328

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SILT 63

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 12 MSE= 13.23075
Critical Value of Studentized Range= 3.773

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
6 - 5	-5.109		3.750		12.609	
6 - 1	-1.527		5.990		13.507	
5 - 6	-12.609		-3.750		5.109	
5 - 1	-4.148		2.240		8.628	
1 - 6	-13.507		-5.990		1.527	
1 - 5	-8.628		-2.240		4.148	

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 25

NOTE: Due to missing values, only 11 observations can be used in this analysis.

Dependent Variable: SILT 50

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	115.51189697	6.62	0.0188
Error	7	40.72666667		
Corrected Total	10	156.23856364		
R-Square		C.V.		
Mean		SILT 50		

0.739330 4.089771 58.97818182

Source	DF	Type I SS	F Value	Pr > F
METHOD	3	115.51189697	6.62	0.0188
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	115.51189697	6.62	0.0188

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 26

NOTE: Due to missing values, only 11 observations can be used in this analysis.

Dependent Variable: SILT 50

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	116.06848485	100.85	0.0001
Error	7	2.68553333		
Corrected Total	10	118.75401818		
Mean				
R-Square			C.V.	SILT 50
		0.977386	0.867829	71.37272727
Source	DF	Type I SS	F Value	Pr > F
METHOD	3	116.06848485	100.85	0.0001
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	116.06848485	100.85	0.0001

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SILT 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 7 MSE= 5.818095
Critical Value of Studentized Range= 4.681

Comparisons significant at the 0.05 level are indicated by '***'.

METHOD Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
3 - 2	-4.853	1.667	8.186		
3 - 4	-3.153	3.367	9.886		
3 - 1	2.148	9.437	16.725		***
2 - 3	-8.186	-1.667	4.853		
2 - 4	-4.819	1.700	8.219		
2 - 1	0.481	7.770	15.059		***
4 - 3	-9.886	-3.367	3.153		
4 - 2	-8.219	-1.700	4.819		
4 - 1	-1.219	6.070	13.359		
1 - 3	-16.725	-9.437	-2.148		***
1 - 2	-15.059	-7.770	-0.481		***
1 - 4	-13.359	-6.070	1.219		

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 26

NOTE: Due to missing values, only 10 observations can be used in this analysis.

Dependent Variable: SILT 50

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	119.26362667	42.49	0.0002
Error	6	5.61413333		
Corrected Total	9	124.87776000		
Mean				
R-Square		C.V.		SILT 50
	0.955043	9.141090	10.58200000	

Source	DF	Type I SS	F Value	Pr > F
METHOD	3	119.26362667	42.49	0.0002
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	119.26362667	42.49	0.0002

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SILT 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 7 MSE= 0.383648

Critical Value of Studentized Range= 4.681

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
4 - 3	1.693	3.367	3.367	5.041	***
4 - 2	2.726	4.400	4.400	6.074	***
4 - 1	7.895	9.767	9.767	11.638	***
3 - 4	-5.041	-3.367	-3.367	-1.693	***
3 - 2	-0.641	1.033	1.033	2.707	***
3 - 1	4.528	6.400	6.400	8.272	***
2 - 4	-6.074	-4.400	-4.400	-2.726	***
2 - 3	-2.707	-1.033	-1.033	0.641	***
2 - 1	3.495	5.367	5.367	7.238	***
1 - 4	-11.638	-9.767	-9.767	-7.895	***
1 - 3	-8.272	-6.400	-6.400	-4.528	***
1 - 2	-7.238	-5.367	-5.367	-3.495	***

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SILT 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 6 MSE= 0.935689
Critical Value of Studentized Range= 4.896

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 2	-2.523	0.533	3.590	-2.523	0.533	3.590
4 - 1	3.567	6.623	9.680	3.567	6.623	9.680
4 - 3	4.666	7.400	10.134	4.666	7.400	10.134
2 - 4	-3.590	-0.533	2.523	-3.590	-0.533	2.523
2 - 1	2.741	6.090	9.439	2.741	6.090	9.439
2 - 3	3.810	6.867	9.923	3.810	6.867	9.923
1 - 4	-9.680	-6.623	-3.567	-9.680	-6.623	-3.567
1 - 2	-9.439	-6.090	-2.741	-9.439	-6.090	-2.741
1 - 3	-2.280	0.777	3.833	-2.280	0.777	3.833
3 - 4	-10.134	-7.400	-4.666	-10.134	-7.400	-4.666
3 - 2	-9.923	-6.867	-3.810	-9.923	-6.867	-3.810
3 - 1	-3.833	-0.777	2.280	-3.833	-0.777	2.280

8. Particle size distribution SAND

units: %
reported by 11 laboratories

TABLE I : Particle size distribution sand - Overview of reported data

labcode	samples reported	number of replicates	fraction
1	A1 B1 B2	3	50-2000µm
2			
3			
4	B1 B2	B1: 2 B2: 1	63-2000µm
5			
6			
7	A1 B1 B2	3	50-2000µm
8	A1 B1 B2	1	63-2000µm
9	A1 B1 B2	3	63-2000µm
10	A1 B1 B2	3	63-2000µm
11			
12	A1 B1 B2	3	63-2000µm
13			
14	A1 B1 B2	3	63-2000µm
15	A1 B1 B2	2	50-2000µm
16			
17			
18			
19	A1 B1 B2	3	63-2000µm
20			
21	A1 B1 B2	3	63-2000µm
22			

TABLE II : Particle size distribution sand - Summary method descriptions

method	description
1	reference, ISRIC 1987
2	hydrometer, without pre-treatment
3	AFNOR X31-107
4	areometer
5	determination without destruction of carbonates and organic matter
6	unspecified

TABLE IIIa : Particle size distribution sand - Descriptive statistics

sample A1 - fraction 50-2000µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
2	2	3	20.40	20.73	21.40	0.58	2.79
7	3	3	14.90	14.93	15.00	0.06	0.39
15	1	2	24.88	24.98	25.08	0.14	0.57

sample A1 - fraction 63-2000µm - national/reference methods

labcode	meth	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5							
6							
7							
8	6	1	19.00	19.00			
9	5	3	17.00	17.00	17.00	0.00	0.00
10	1	3	15.40	16.93	19.30	2.08	12.28
11							
12	1	3	20.30	21.60	23.40	1.61	7.45
13							
14	4	3	18.20	19.13	20.40	1.14	5.94
15							
16							
17							
18							
19	5	3	17.80	17.80			
20							
21	5	3	14.50	15.07	15.60	0.55	3.66
22							

sample B1 - fraction 50-2000µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
2	2	3	10.20	10.87	12.20	1.16	10.63
7	3	3	4.50	4.87	5.60	0.64	13.05
15	1	2	8.75	9.49	10.23	1.05	11.03

sample B1 - fraction 63-2000µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5	6	1		4.00			
6							
7	3						
8	6	1		2.40			
9	5	3	2.00	2.00	2.00	0.00	0.00
10	1	3	3.30	4.63	6.40	1.60	34.42
11							
12	1	3	52.70	53.27	53.90	0.60	1.13
13							
14	4	3	1.60	1.77	1.90	0.15	8.65
15							
16							
17							
18							
19	5	3		7.40			
20							
21	5	3	1.90	2.03	2.30	0.23	11.36
22							

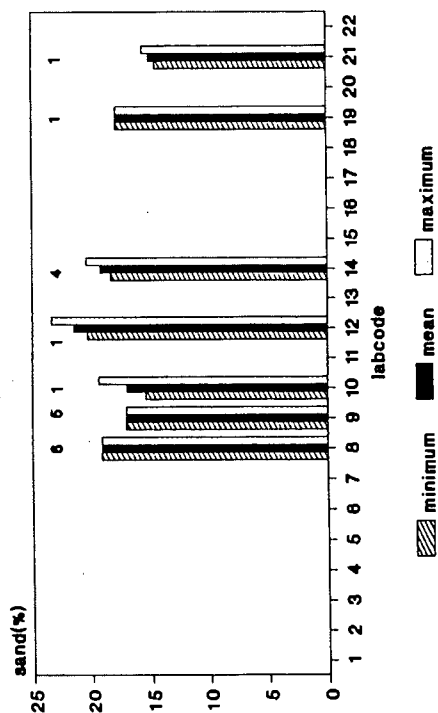
sample B2 - fraction 50-2000µm - national/reference methods

labcode	method	repl	minimum	mean	maximum	std	cv
2	2	2	85.30	86.30	87.30	1.41	1.64
7	3	3	83.60	84.83	86.30	1.37	1.61
15	1	2	85.88	87.09	88.29	1.70	1.96

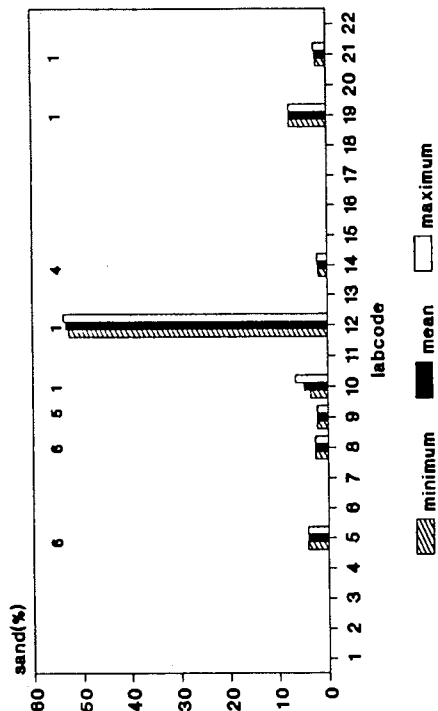
sample B2 - fraction 63-2000µm - national/reference methods

labcode	meth	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4							
5	6	1		81.50			
6							
7	3						
8	6	1		83.80			
9	5	3	75.00	76.00	77.00	1.00	1.32
10	1	3	78.50	79.27	80.50	1.08	1.36
11							
12	1	3	83.40	83.63	84.10	0.40	0.48
13							
14	4	3	81.90	82.20	82.40	0.27	0.32
15							
16							
17							
18							
19	5	3		77.90			
20							
21	5	3	90.80	92.07	93.40	1.30	1.41
22							

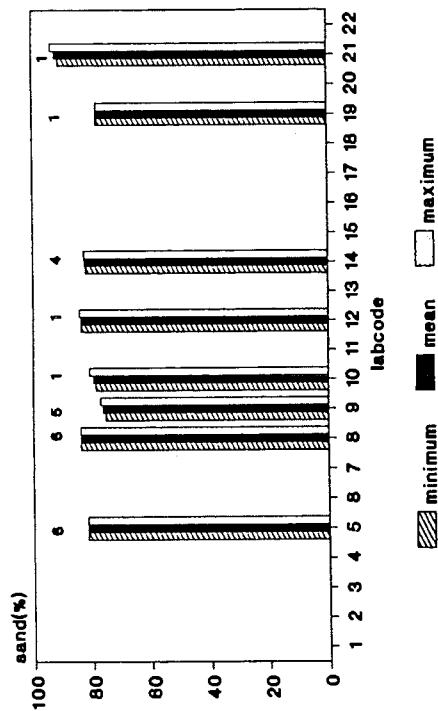
sand sample A1
fraction 63-2000µm



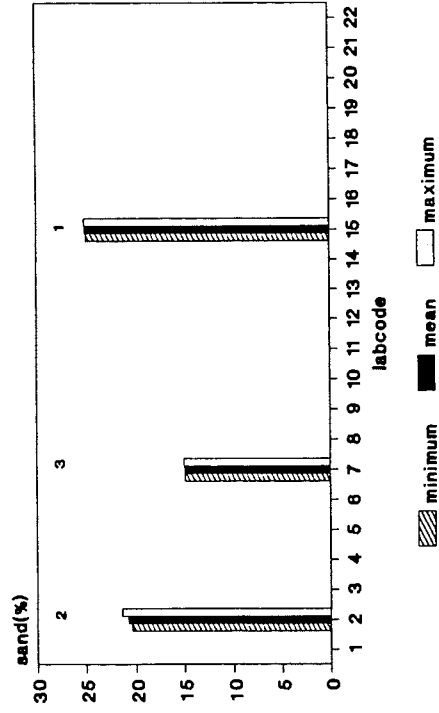
sand sample B1
fraction 63-2000µm



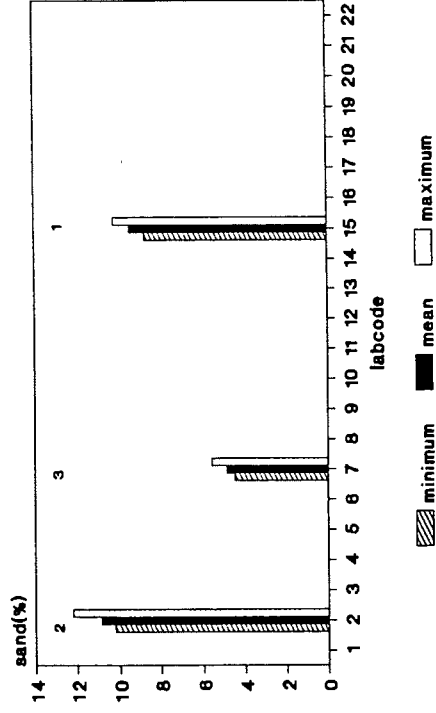
sand sample B2
fraction 63-2000µm



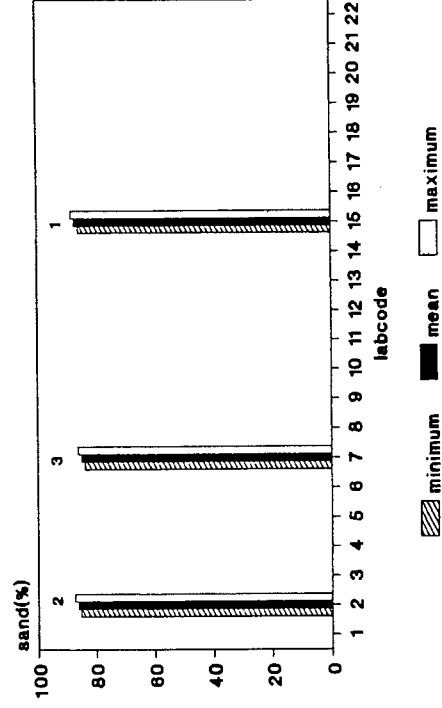
sand sample A1
fraction 50-2000µm



sand sample B1
fraction 50-2000µm



sand sample B2
fraction 50-2000µm



SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 25

NOTE: Due to missing values, only 17 observations can be used in this analysis.

Dependent Variable: SAND 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	8.04933333	0.41	0.7482
Error	13	84.97066667		
Corrected Total	16	93.02000000		
R-Square C.V. SAND 63 Mean				
		14.20334	18.00000000	
		0.086533		
Source	DF	Type I SS	F Value	Pr > F
METHOD	3	8.04933333	0.41	0.7482
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	8.04933333	0.41	0.7482

TABLE IV : Particle size distribution Sand - Total mean and normal distribution

fraction 63-2000µm

sample	method	repl	mean	std	normal	prob<W
A1	1	10	17.86	3.03	0.91	0.28
A1	4	3	19.13	1.14	0.94	0.51
A1	5	3	17.00	0.00		
A1	6	1	19.00			
B1	1	10	18.72	23.91	0.66	0.00
B1	4	3	1.77	0.15	0.96	0.64
B1	5	3	2.00	0.00		
B1	6	2	3.20	1.13	1.00	1.00
B2	1	10	84.28	5.82	0.88	0.12
B2	4	3	82.20	0.27	0.89	0.36
B2	5	3	76.00	1.00	1.00	0.99
B2	6	2	82.65	1.63	1.00	1.00

fraction 50-2000µm

sample	method	repl	mean	std	normal	prob<W
A1	1	2	24.98	0.14	1.00	1.00
A1	2	3	20.73	0.58	0.75	0.00
A1	3	3	14.93	0.06	0.75	0.00
B1	1	2	9.49	1.05	1.00	1.00
B1	2	3	10.87	1.16	0.75	0.00
B1	3	3	4.87	0.64	0.75	0.00
B2	1	2	87.09	1.70	1.00	1.00
B2	2	2	86.30	1.41	1.00	1.00
B2	3	3	84.83	1.37	0.98	0.72

Remark:

Labcode 19 reported mean values for 3 replicates.
To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.
So, the "repl" values in this table for method 5 are different from the real number of replicates.

SAMPLE=B1

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Tukey's Studentized Range (HSD) Test for variable: SAND 63

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 13 MSE= 6.536205
Critical Value of Studentized Range= 4.151

Comparisons significant at the 0.05 level are indicated by '****'.

NOTE: Due to missing values, only 18 observations can be used in this analysis.

Dependent Variable: SAND 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	1213.78233333	1.10	0.3816
Error	14	5146.44266667		
Corrected Total	17	6360.22500000		

R-Square C.V. SAND 63 Mean

0.190840 168.4303 11.38333333

Source	DF	Type I SS	F Value	Pr > F
METHOD	3	1213.78233333	1.10	0.3816
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	1213.78233333	1.10	0.3816

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 25

NOTE: Due to missing values, only 18 observations can be used in this analysis.

Dependent Variable: SAND 63

Source	DF	Sum of Squares	F Value	Pr > F
Model	3	158.45511111	2.38	0.1130
Error	14	310.08100000		
Corrected Total	17	468.53611111		

SAND 63 Mean

C.V.

R-Square

0.338192

5.713376

82.37222222

Source	DF	Type I SS	F Value	Pr > F
METHOD	3	158.45511111	2.38	0.1130
Source	DF	Type III SS	F Value	Pr > F
METHOD	3	158.45511111	2.38	0.1130

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SAND 63

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 14 MSE= 367.603

Critical Value of Studentized Range= 4.111

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 6	-27.646	15.520	15.520	58.686	
1 - 5	-19.964	16.720	16.720	53.404	
1 - 4	-19.731	16.953	16.953	53.638	
6 - 1	-58.686	-15.520	-15.520	27.646	
6 - 5	-49.672	1.200	1.200	52.072	
6 - 4	-49.439	1.433	1.433	52.305	
5 - 1	-53.404	-16.720	-16.720	19.964	
5 - 6	-52.072	-1.200	-1.200	49.672	
5 - 4	-45.268	0.233	0.233	45.735	
4 - 1	-53.638	-16.953	-16.953	19.731	
4 - 6	-52.305	-1.433	-1.433	49.439	
4 - 5	-45.735	-0.233	-0.233	45.268	

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SAND 63

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 14 MSE= 22.14864

Critical Value of Studentized Range= 4.111

Comparisons significant at the 0.05 level are indicated by '****'.

General Linear Models Procedure

Class Level Information

Class Levels Values

METHOD 6 1 2 3 4 5 6

Number of observations in by group = 25

METHOD Comparison		Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
1	- 6	-8.966	1.630	12.226
1	- 4	-6.925	2.080	11.085
1	- 5	-0.725	8.280	17.285
6	- 1	-12.226	-1.630	8.966
6	- 4	-12.037	0.450	12.937
6	- 5	-5.837	6.650	19.137
4	- 1	-11.085	-2.080	6.925
4	- 6	-12.937	-0.450	12.037
4	- 5	-4.969	6.200	17.369
5	- 1	-17.285	-8.280	0.725
5	- 6	-19.137	-6.650	5.837
5	- 4	-17.369	-6.200	4.969

Dependent Variable: SAND 50

Source DF Sum of Squares F Value Pr > F

Model 2 127.07226667 458.19 0.0001

Error 5 0.69333333

Corrected Total 7 127.76560000

R-Square 0.994573

Mean 1.897960

C.V. 19.62000000

Source DF Type I SS F Value Pr > F

METHOD 2 127.07226667 458.19 0.0001

Source DF Type III SS F Value Pr > F

METHOD 2 127.07226667 458.19 0.0001

-----SAMPLE=A1-----

-----SAMPLE=B1-----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SAND 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 5 MSE= 0.138667
Critical Value of Studentized Range= 4.600

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means		Lower Confidence Limit	Upper Confidence Limit
1 - 2	3.141	4.247	4.247	5.352	***	***
1 - 3	8.941	10.047	10.047	11.152	***	***
2 - 1	-5.352	-4.247	-4.247	-3.141	***	***
2 - 3	4.811	5.800	5.800	6.789	***	***
3 - 1	-11.152	-10.047	-10.047	-8.941	***	***
3 - 2	-6.789	-5.800	-5.800	-4.811	***	***

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 26

NOTE: Due to missing values, only 8 observations can be used in this analysis.

Dependent Variable: SAND 50

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	57.95281667	31.71	0.0014
Error	5	4.56853333		
Corrected Total	7	62.52135000		
R-Square	C.V.			
Mean	SAND 50			

0.926928	11.55491	8.27250000

Source	DF	Type I SS	F Value	Pr > F
METHOD	2	57.95281667	31.71	0.0014
Source	DF	Type III SS	F Value	Pr > F
METHOD	2	57.95281667	31.71	0.0014

----- SAMPLE=B2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METHOD	6	1 2 3 4 5 6

Number of observations in by group = 25

NOTE: Due to missing values, only 7 observations can be used in this analysis.

Dependent Variable: SAND 50				
Source	DF	Sum of Squares	F Value	Pr > F
Model	2	6.54165476	1.52	0.3236
Error	4	8.63071667		
Corrected Total	6	15.17237143		
R-Square C.V. SAND 50				
Mean				
		1.710102		85.89571429

Source	DF	Type I SS	F Value	Pr > F
METHOD	2	6.54165476	1.52	0.3236
Source	DF	Type III SS	F Value	Pr > F
METHOD	2	6.54165476	1.52	0.3236

----- SAMPLE=B1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SAND 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 5 MSE= 0.913707
Critical Value of Studentized Range= 4.600

Comparisons significant at the 0.05 level are indicated by '***'.

METHOD Comparison	Simultaneous Lower Confidence Limit		Simultaneous Upper Confidence Limit	
	Difference Between Means			
2 - 1	-1.462	1.377	4.215	***
2 - 3	3.461	6.000	8.539	***
1 - 2	-4.215	-1.377	1.462	***
1 - 3	1.785	4.623	7.462	***
3 - 2	-8.539	-6.000	-3.461	***
3 - 1	-7.462	-4.623	-1.785	***

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: SAND 50

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 4 MSE= 2.157679
Critical Value of Studentized Range= 5.040

Comparisons significant at the 0.05 level are indicated by '****'.

METHOD Comparison	Simultaneous		Difference		Simultaneous
	Lower Limit	Confidence	Between Means	Upper Limit	Confidence
1 - 2	-4.450		0.785	6.020	
1 - 3	-2.527		2.252	7.031	
2 - 1	-6.020		-0.785	4.450	
2 - 3	-3.312		1.467	6.246	
3 - 1	-7.031		-2.252	2.527	
3 - 2	-6.246		-1.467	3.312	

9. EXCHANGEABLE ACIDITY

units: cmol+/kg
mandatory for samples B1 and B2
reported by 20 laboratories

TABLE I : Exchangeable acidity - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2	B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5	B1 B2	1 not reported	N
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	B1 B2	3	R
10	B1 B2	3	R
11	B1 B2	3	R
12	B1 B2	B1:7 B2:5	N
13	A1 B1 B2	3	N
14	A1 B1 B2	5	R
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	2	N
19	A1 B1 B2	3	R
20			
21	A1 B1 B2	3	N/R
22	B1 B2	3	N

TABLE II : Exchangeable acidity - Summary method descriptions

method	description
1	reference
2	total CEC - sum(Ca2+, Mg2+, K+)
3	1M NH4NO3, titration up to pH 7
4	1M KCl, titration up to pH 8.2
5	1N KCl, titration up to rose colour
6	CEC - sum basic cations
7	0.1M BaCl2, titration up to pH 7
8	sum acid cations
9	1M KCl, titrate up to pH 8.2; 4% NaF, titrate up to pH 8.2
10	1M HCl, titrate up to pH 7.8
11	cobaltihexamine 0.05M 2.5:50
12	0.5M Ba(COOCH3)2
13	unspecified

TABLE IIlb : Exchangeable acidity - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
14	5	0.006	0.007	0.008	0.001	13.552
19	3		0.430			
21	3	3.770	4.393	5.270	0.781	17.787

sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	48.770	49.130	49.550	0.335	0.682
5						
6	2	3.620	3.670	3.720	0.071	1.927
7	3	4.020	4.087	4.180	0.083	2.038
8	3	2.330	2.413	2.540	0.112	4.620
9	3	5.688	5.769	5.811	0.070	1.216
10	3	4.260	4.290	4.320	0.030	0.699
11	3	7.200	7.317	7.500	0.161	2.197
12						
13						
14	5	3.919	3.986	4.072	0.056	1.394
15	3	4.270	4.403	4.540	0.135	3.067
16	3	4.120	4.157	4.180	0.032	0.773
17						
18						
19	3	41.370	41.370	41.370		
20						
21	3	8.660	9.583	10.600	0.973	10.157
22						

sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	24.670	25.550	26.270	0.670	2.620
5						
6	2	1.390	1.460	1.530	0.099	6.780
7	3	2.240	2.267	2.300	0.031	1.348
8	3	1.330	1.510	1.650	0.164	10.842
9	3	3.914	4.069	4.178	0.138	3.392
10	3	2.790	2.843	2.870	0.046	1.624
11	3	3.650	3.800	4.000	0.180	4.744
12						
13						
14	5	2.350	2.464	2.675	0.128	5.195
15	3	2.420	2.613	2.710	0.167	6.407
16	3	2.580	2.610	2.630	0.026	1.014
17						
18						
19	3		26.590			
20						
21	3	1.910	1.973	2.060	0.078	3.936
22						

TABLE IIIa : Exchangeable acidity - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	3	5	0.000	0.000	0.000	0.000	
14	7	5	0.000	0.000	0.000	0.000	
21	12	3	0.000	0.000	0.000	0.000	

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	3	5	4.070	4.263	4.470	0.150	3.521
13	6	3	7.520	9.063	10.990	1.766	19.490

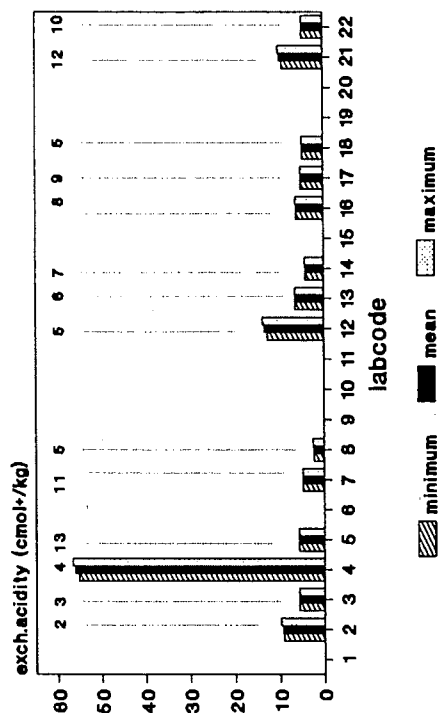
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	9.090	9.380	9.790	0.365	3.892
3	3	5	5.720	5.766	5.810	0.030	0.520
4	4	4	55.340	56.185	56.750	0.625	1.113
5	13	1	5.760	5.760	5.760		
6							
7	11	3	4.700	4.740	4.800	0.053	1.116
8	5	3	2.260	2.343	2.470	0.112	4.758
9							
10							
11							
12	5	7	12.600	13.257	13.700	0.424	3.196
13	6	3	6.390	6.453	6.500	0.057	0.881
14	7	5	4.066	4.105	4.140	0.036	0.868
15							
16	8	3	6.210	6.233	6.250	0.021	0.334
17	9	3	5.150	5.197	5.230	0.042	0.801
18	5	2	4.689	4.730	4.771	0.058	1.226
19							
20							
21	12	3	9.340	9.840	10.200	0.447	4.540
22	10	3	4.770	4.800	4.820	0.026	0.551

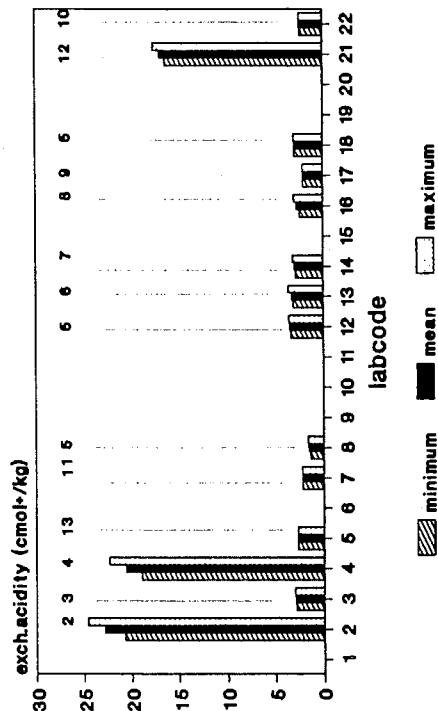
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	20.790	22.770	24.660	1.937	8.505
3	3	5	2.900	2.959	3.040	0.060	2.027
4	4	4	18.950	20.635	22.340	1.684	8.161
5	13	1	2.750	2.750	2.750		
6							
7	11	3	2.220	2.227	2.240	0.012	0.519
8	5	3	1.320	1.513	1.640	0.170	11.240
9							
10							
11							
12	5	5	3.400	3.520	3.600	0.084	2.377
13	6	3	3.110	3.297	3.640	0.298	9.031
14	7	5	2.852	2.953	3.185	0.137	4.651
15							
16	8	3	2.490	2.810	3.040	0.286	10.172
17	9	3	2.080	2.100	2.130	0.026	1.260
18	5	2	2.965	3.011	3.056	0.064	2.137
19							
20							
21	12	3	16.300	16.900	17.600	0.656	3.880
22	10	3	2.410	2.427	2.450	0.021	0.858

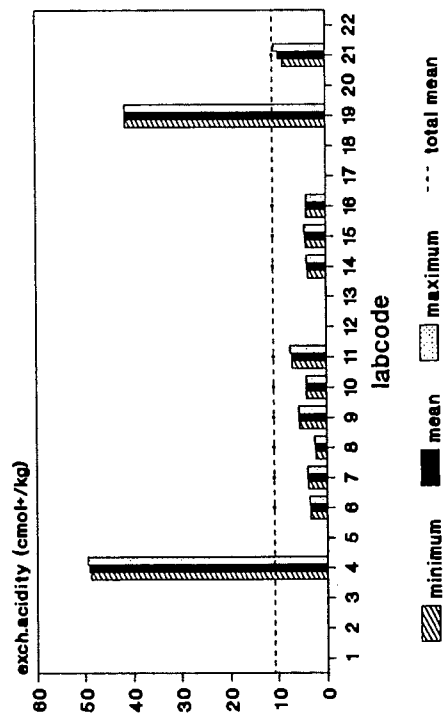
Exch.acidity sample B1
national methods



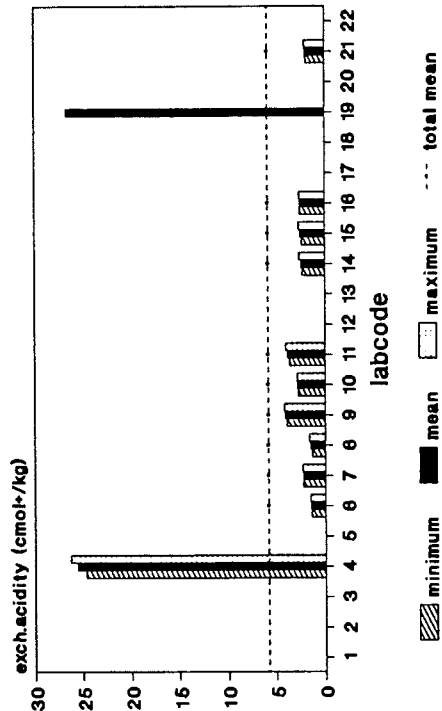
Exch.acidity sample B2
national methods



exch.acidity sample B1
reference method



exch.acidity sample B2
reference method



----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	13	1 10 11 12 13 2 3 4 5 6 7 8 9

Number of observations in by group = 80

Dependent Variable: EXCH.ACIDITY

Source	DF	Sum of Squares	F Value	Pr > F
Model	12	8975.20648449	6.04	0.0001
Error	67	8291.69543200		
Corrected Total	79	17266.90191649		
Mean				
	R-Square	C.V.	EXCH.ACIDITY	
	0.519793	99.19070	11.21536250	
Source	DF	Type I SS	F Value	Pr > F
METH	12	8975.20648449	6.04	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	12	8975.20648449	6.04	0.0001

TABLE IV : Exchangeable acidity - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	1.516	2.197	0.709	0.002
A2	3	1	4.263			
A2	6	3	9.063	1.766	0.965	0.639
B1	1	36	10.867	15.112	0.516	0.000
B1	2	3	9.380	0.365	0.919	0.449
B1	3	1	5.766			
B1	4	4	56.185	0.625	0.929	0.574
B1	5	12	9.108	5.198	0.741	0.002
B1	6	3	6.453	0.057	0.936	0.510
B1	7	5	4.105	0.036	0.804	0.009
B1	8	3	6.233	0.021	0.923	0.463
B1	9	3	5.197	0.042	0.923	0.463
B1	10	3	4.800	0.026	0.893	0.363
B1	11	3	4.740	0.053	0.893	0.363
B1	12	3	9.840	0.447	0.926	0.475
B1	13	1	5.760			
B2	1	36	5.808	8.163	0.498	0.000
B2	2	3	22.770	1.937	0.998	0.923
B2	3	1	2.959			
B2	4	4	20.635	1.684	0.872	0.301
B2	5	10	2.817	0.927	0.780	0.009
B2	6	3	3.297	0.298	0.792	0.096
B2	7	5	2.953	0.137	0.802	0.083
B2	8	3	2.810	0.286	0.926	0.473
B2	9	3	2.100	0.026	0.893	0.363
B2	10	3	2.427	0.021	0.923	0.463
B2	11	3	2.227	0.012	0.750	0.000
B2	12	3	16.900	0.656	0.983	0.747
B2	13	1	2.750			

Remark:

Labcode 3 and labcode 19 reported mean values for 3 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for methods 1 and 3 are different from the real number
of replicates.

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable:

EXCH. ACIDITY

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 67 MSE= 123.7566

Critical Value of Studentized Range= 4.858

comparisons significant at the 0.05 level are indicated by '***'.

Tukey's Studentized Range (HSD) Test for variable: EXCH.ACIDITY										
E: This test controls the type I experimentwise error rate.										
Alpha= 0.05 Confidence= 0.95 df= 67 MSE= 123.7566										
Critical Value of Studentized Range= 4.858										
isons significant at the 0.05 level are indicated by '***'.										
Simultaneous					Simultaneous					
METH	Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	METH	Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
4	- 1	25.177	45.318	65.458	***	2	- 4	-75.991	-46.805	***
4	- 12	17.159	46.345	75.531	***	2	- 1	-24.451	-1.487	***
4	- 2	17.619	46.805	75.991	***	2	- 12	-31.661	-0.460	***
4	- 5	25.015	47.077	69.140	***	2	- 5	-24.394	0.272	***
4	- 6	20.546	49.732	78.918	***	2	- 6	-28.275	2.927	***
4	- 8	20.766	49.952	79.138	***	2	- 8	-28.055	3.147	***
4	- 3	7.695	50.419	93.143	***	2	- 3	-40.511	3.614	***
4	- 13	7.701	50.425	93.149	***	2	- 13	-40.505	3.620	***
4	- 9	21.802	50.988	80.174	***	2	- 9	-27.018	4.183	***
4	- 10	22.199	51.385	80.571	***	2	- 10	-26.621	4.580	***
4	- 11	22.259	51.445	80.631	***	2	- 11	-26.561	4.640	***
4	- 7	26.446	52.080	77.714	***	2	- 7	-22.632	5.275	***
1	- 4	-65.458	-45.318	-25.177	***	5	- 4	-69.140	-47.077	***
1	- 12	-21.936	1.027	23.991	***	5	- 1	-14.498	-1.760	***
1	- 2	-21.476	1.487	24.451	***	5	- 12	-25.399	-0.732	***
1	- 5	-10.978	1.760	14.498	***	5	- 2	-24.939	-0.272	***
1	- 6	-18.550	4.414	27.377	***	5	- 6	-22.013	2.654	***
1	- 8	-18.330	4.634	27.597	***	5	- 8	-21.793	2.874	***
1	- 3	-33.640	5.101	43.842	***	5	- 3	-36.432	3.342	***
1	- 13	-33.634	5.107	43.848	***	5	- 13	-36.426	3.348	***
1	- 9	-17.293	5.671	28.634	***	5	- 9	-20.756	3.911	***
1	- 10	-16.896	6.067	29.031	***	5	- 10	-20.359	4.307	***
1	- 11	-16.836	6.127	29.091	***	5	- 11	-20.299	4.368	***
1	- 7	-11.476	6.762	25.000	***	5	- 7	-15.338	5.003	***
12	- 4	-75.531	-46.345	-17.159	***	6	- 4	-78.918	-49.732	***
12	- 1	-30.991	-1.027	21.936	***	6	- 1	-27.377	-4.414	***
12	- 2	-30.741	0.460	31.661	***	6	- 12	-34.588	-3.387	***
12	- 5	-23.934	0.732	25.399	***	6	- 2	-34.128	-2.927	***
12	- 6	-27.815	3.387	34.588	***	6	- 5	-27.321	-2.654	***
12	- 8	-27.595	3.607	34.808	***	6	- 8	-30.981	0.220	***
12	- 3	-40.051	4.074	48.199	***	6	- 3	-43.438	0.687	***
12	- 13	-40.045	4.080	48.205	***	6	- 13	-43.432	0.693	***
12	- 9	-26.558	4.643	35.845	***	6	- 9	-29.945	1.257	***
12	- 10	-26.161	5.040	36.241	***	6	- 10	-29.548	1.653	***
12	- 11	-26.101	5.100	36.301	***	6	- 11	-29.488	1.713	***
12	- 7	-22.322	5.725	32.542	***	6	- 7	-25.559	2.348	***
12	- 4	-75.531	-46.345	-17.159	***	8	- 4	-79.138	-49.952	***
12	- 1	-30.991	-1.027	21.936	***	8	- 1	-27.597	-4.634	***
12	- 2	-30.741	0.460	31.661	***	8	- 12	-34.808	-3.607	***
12	- 5	-23.934	0.732	25.399	***	8	- 2	-34.348	-2.147	***
12	- 6	-27.815	3.387	34.588	***	8	- 5	-27.541	-2.874	***
12	- 8	-27.595	3.607	34.808	***	8	- 6	-31.421	-0.220	***
12	- 3	-40.051	4.074	48.199	***	8	- 3	-43.658	0.467	***
12	- 13	-40.045	4.080	48.205	***	8	- 13	-43.652	0.473	***
12	- 9	-26.558	4.643	35.845	***	8	- 9	-43.652	0.473	***
12	- 10	-26.161	5.040	36.241	***	8	- 10	-43.652	0.473	***
12	- 11	-26.101	5.100	36.301	***	8	- 11	-43.652	0.473	***
12	- 7	-22.322	5.725	32.542	***	8	- 7	-30.165	1.037	***

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
10 - 6	-32.855	-1.653	-1.653	-32.855	29.548
10 - 8	-32.635	-1.433	-1.433	-32.635	29.768
10 - 3	-45.091	-0.966	-0.966	-45.091	43.159
10 - 13	-45.085	-0.960	-0.960	-45.085	43.165
10 - 9	-31.598	-0.397	-0.397	-31.598	30.805
10 - 11	-31.141	0.060	0.060	-31.141	31.261
10 - 7	-27.212	0.695	0.695	-27.212	28.602
11 - 4	-80.631	-51.445	-51.445	-80.631	-22.259
11 - 1	-29.091	-6.127	-6.127	-29.091	16.836
11 - 12	-36.301	-5.100	-5.100	-36.301	26.101
11 - 2	-35.841	-4.640	-4.640	-35.841	26.561
11 - 5	-29.034	-4.368	-4.368	-29.034	20.299
11 - 6	-32.915	-1.713	-1.713	-32.915	29.488
11 - 8	-32.695	-1.493	-1.493	-32.695	29.708
11 - 3	-45.151	-1.026	-1.026	-45.151	43.099
11 - 13	-45.145	-1.020	-1.020	-45.145	43.105
11 - 9	-31.658	-0.457	-0.457	-31.658	30.745
11 - 10	-31.261	-0.060	-0.060	-31.261	31.141
11 - 7	-27.272	0.635	0.635	-27.272	28.542
7 - 4	-77.714	-52.080	-52.080	-77.714	-26.446
7 - 1	-25.000	-6.762	-6.762	-25.000	11.476
7 - 12	-33.642	-5.735	-5.735	-33.642	22.172
7 - 2	-33.182	-5.275	-5.275	-33.182	22.632
7 - 5	-25.343	-5.003	-5.003	-25.343	15.338
7 - 6	-30.256	-2.348	-2.348	-30.256	25.559
7 - 8	-30.036	-2.128	-2.128	-30.036	25.779
7 - 3	-43.522	-1.661	-1.661	-43.522	40.200
7 - 13	-43.516	-1.655	-1.655	-43.516	40.206
7 - 9	-28.999	-1.092	-1.092	-28.999	26.816
7 - 10	-28.602	-0.695	-0.695	-28.602	27.212
7 - 11	-28.542	-0.635	-0.635	-28.542	27.272

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
8 - 10	-29.768	1.433	1.433	-29.768	32.635
8 - 11	-29.708	1.493	1.493	-29.708	32.695
8 - 7	-25.779	2.128	2.128	-25.779	30.036
3 - 4	-93.143	-50.419	-50.419	-93.143	-7.695
3 - 1	-43.842	-5.101	-5.101	-43.842	33.640
3 - 12	-48.199	-4.074	-4.074	-48.199	40.051
3 - 2	-47.739	-3.614	-3.614	-47.739	40.511
3 - 5	-43.115	-3.342	-3.342	-43.115	36.432
3 - 6	-44.813	-0.687	-0.687	-44.813	43.438
3 - 8	-44.593	-0.467	-0.467	-44.593	43.658
3 - 13	-54.036	0.006	0.006	-54.036	54.048
3 - 9	-43.556	0.569	0.569	-43.556	44.695
3 - 10	-43.159	0.966	0.966	-43.159	45.091
3 - 11	-43.099	1.026	1.026	-43.099	45.151
3 - 7	-40.200	1.661	1.661	-40.200	43.522
13 - 4	-93.149	-50.425	-50.425	-93.149	-7.701
13 - 1	-43.848	-5.107	-5.107	-43.848	33.634
13 - 12	-48.205	-4.080	-4.080	-48.205	40.045
13 - 2	-47.745	-3.620	-3.620	-47.745	40.505
13 - 5	-43.121	-3.348	-3.348	-43.121	36.426
13 - 6	-44.819	-0.693	-0.693	-44.819	43.432
13 - 8	-44.599	-0.473	-0.473	-44.599	43.652
13 - 3	-54.048	-0.006	-0.006	-54.048	54.036
13 - 9	-43.562	0.563	0.563	-43.562	44.689
13 - 10	-43.165	0.960	0.960	-43.165	45.085
13 - 11	-43.105	1.020	1.020	-43.105	45.145
13 - 7	-40.206	1.655	1.655	-40.206	43.516
9 - 4	-80.174	-50.988	-50.988	-80.174	-21.802
9 - 1	-28.634	-5.671	-5.671	-28.634	17.293
9 - 12	-35.845	-4.643	-4.643	-35.845	26.558
9 - 2	-35.385	-4.183	-4.183	-35.385	27.018
9 - 5	-28.578	-3.911	-3.911	-28.578	20.756
9 - 6	-32.458	-1.257	-1.257	-32.458	29.945
9 - 8	-32.238	-1.037	-1.037	-32.238	30.165
9 - 3	-44.695	-0.569	-0.569	-44.695	43.556
9 - 13	-44.689	-0.563	-0.563	-44.689	43.562
9 - 10	-30.805	0.397	0.397	-30.805	31.598
9 - 11	-30.745	0.457	0.457	-30.745	31.658
9 - 7	-26.816	1.092	1.092	-26.816	28.999
10 - 4	-80.571	-51.385	-51.385	-80.571	-22.199
10 - 1	-29.031	-6.067	-6.067	-29.031	16.896
10 - 12	-36.241	-5.040	-5.040	-36.241	26.161
10 - 2	-35.781	-4.580	-4.580	-35.781	26.621
10 - 5	-28.974	-4.307	-4.307	-28.974	20.359

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Class Levels Values

Tukey's Studentized Range (HSD) Test for variable:
EXCH.ACIDITY

Number of observations in by group = 78

NOTE: This test controls the type I experimentwise error rate.
Alpha= 0.05 Confidence= 0.95 df= 65 MSE= 36.26551
Critical Value of Studentized Range= 4.863

Dependent Variable: EXCH.ACIDITY

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous										Simultaneous									
Source		Sum of Squares		F Value		Pr > F		METH Comparison		Lower Confidence Limit		Between Means		Upper Confidence Limit					
Model		2393.41743866		5.50		0.0001		2 - 4		-13.682		2.135		17.952					
Error		2357.25812617						2 - 12		-11.039		5.870		22.779					
Corrected Total		4750.67556483						2 - 1		4.517		16.962		29.407					
R-Square		C.V.		EXCH.ACIDITY				2 - 6		2.564		19.473		36.382					
Mean		94.34815		6.38283333				2 - 3		-4.102		19.811		43.724					
R-Square		0.503806						2 - 7		4.694		19.817		34.941					
Type I SS				6.321				19.953		33.585									
Type III SS		94.34815		6.38283333				2 - 5		3.051		19.960		36.869					
Type I SS		94.34815		6.38283333				2 - 8		-3.893		20.020		43.933					
Type III SS		94.34815		6.38283333				2 - 13		3.434		20.343		37.252					
Type I SS		94.34815		6.38283333				2 - 10		3.634		20.543		37.452					
Type III SS		94.34815		6.38283333				2 - 11		3.761		20.670		37.579					
Type I SS		94.34815		6.38283333				2 - 9											
Type III SS		94.34815		6.38283333															
Type I SS		94.34815		6.38283333				4 - 2		-17.952		-2.135		13.682					
Type III SS		94.34815		6.38283333				4 - 12		-12.082		3.735		19.552					
Type I SS		94.34815		6.38283333				4 - 1		3.912		14.827		25.742					
Type III SS		94.34815		6.38283333				4 - 6		1.522		17.338		33.155					
Type I SS		94.34815		6.38283333				4 - 3		-5.477		17.676		40.829					
Type III SS		94.34815		6.38283333				4 - 7		3.790		17.682		31.574					
Type I SS		94.34815		6.38283333				4 - 5		5.566		17.818		30.070					
Type III SS		94.34815		6.38283333				4 - 8		2.008		17.825		33.642					
Type I SS		94.34815		6.38283333				4 - 13		-5.268		17.885		41.038					
Type III SS		94.34815		6.38283333				4 - 10		2.392		18.208		34.025					
Type I SS		94.34815		6.38283333				4 - 11		2.592		18.408		34.225					
Type III SS		94.34815		6.38283333				4 - 9		2.718		18.535		34.352					
Type I SS		94.34815		6.38283333				12 - 2		-22.779		-5.870		11.039					
Type III SS		94.34815		6.38283333				12 - 4		-19.552		-3.735		12.082					
Type I SS		94.34815		6.38283333				12 - 1		-1.353		11.092		23.537					
Type III SS		94.34815		6.38283333				12 - 6		-3.306		13.603		30.512					
Type I SS		94.34815		6.38283333				12 - 3		-9.972		13.941		37.854					
Type III SS		94.34815		6.38283333				12 - 7		-1.176		13.947		29.071					
Type I SS		94.34815		6.38283333				12 - 5		0.451		14.083		27.715					
Type III SS		94.34815		6.38283333				12 - 8		-2.819		14.090		30.999					
Type I SS		94.34815		6.38283333				12 - 13		-9.763		14.150		38.063					
Type III SS		94.34815		6.38283333				12 - 10		-2.436		14.473		31.382					
Type I SS		94.34815		6.38283333				12 - 11		-2.236		14.673		31.582					
Type III SS		94.34815		6.38283333				12 - 9		-2.109		14.800		31.709					

SAMPLE=B2

General Linear Models Procedure

MEVH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
7 - 10	-14.598	0.526	15.650	-14.598	0.526	15.650
7 - 11	-14.398	0.726	15.850	-14.398	0.726	15.850
7 - 9	-14.271	0.853	15.976	-14.271	0.853	15.976
5 - 2	-33.585	-19.953	-6.321	-33.585	-19.953	-6.321
5 - 4	-30.070	-17.818	-5.566	-30.070	-17.818	-5.566
5 - 12	-27.715	-14.083	-0.451	-27.715	-14.083	-0.451
5 - 1	-10.394	-2.991	4.412	-10.394	-2.991	4.412
5 - 6	-14.112	-0.480	13.153	-14.112	-0.480	13.153
5 - 3	-21.862	-0.142	21.578	-21.862	-0.142	21.578
5 - 7	-11.478	-0.136	11.207	-11.478	-0.136	11.207
5 - 8	-13.625	0.007	13.639	-13.625	0.007	13.639
5 - 13	-21.653	0.067	21.787	-21.653	0.067	21.787
5 - 10	-13.242	0.390	14.023	-13.242	0.390	14.023
5 - 11	-13.042	0.590	14.223	-13.042	0.590	14.223
5 - 9	-12.915	0.717	14.349	-12.915	0.717	14.349
8 - 2	-36.869	-19.960	-3.051	-36.869	-19.960	-3.051
8 - 4	-33.642	-17.825	-2.008	-33.642	-17.825	-2.008
8 - 12	-30.999	-14.090	2.819	-30.999	-14.090	2.819
8 - 1	-15.443	-2.998	9.447	-15.443	-2.998	9.447
8 - 6	-17.396	-0.487	16.422	-17.396	-0.487	16.422
8 - 3	-24.062	-0.149	23.764	-24.062	-0.149	23.764
8 - 7	-15.266	-0.143	14.981	-15.266	-0.143	14.981
8 - 5	-13.639	-0.007	13.625	-13.639	-0.007	13.625
8 - 13	-23.853	0.060	23.973	-23.853	0.060	23.973
8 - 10	-16.526	0.383	17.292	-16.526	0.383	17.292
8 - 11	-16.326	0.583	17.492	-16.326	0.583	17.492
8 - 9	-16.199	0.710	17.619	-16.199	0.710	17.619
13 - 2	-43.933	-20.020	3.893	-43.933	-20.020	3.893
13 - 4	-41.038	-17.885	5.268	-41.038	-17.885	5.268
13 - 12	-38.063	-14.150	9.763	-38.063	-14.150	9.763
13 - 1	-24.053	-3.058	17.937	-24.053	-3.058	17.937
13 - 6	-24.459	-0.547	23.366	-24.459	-0.547	23.366
13 - 3	-29.496	-0.209	29.078	-29.496	-0.209	29.078
13 - 7	-22.888	-0.203	22.483	-22.888	-0.203	22.483
13 - 5	-21.787	-0.067	21.653	-21.787	-0.067	21.653
13 - 8	-23.973	-0.060	23.853	-23.973	-0.060	23.853
13 - 10	-23.589	0.323	24.236	-23.589	0.323	24.236
13 - 11	-23.389	0.523	24.436	-23.389	0.523	24.436
13 - 9	-23.263	0.650	24.563	-23.263	0.650	24.563
10 - 2	-37.252	-20.343	-3.434	-37.252	-20.343	-3.434
10 - 4	-34.025	-18.208	-2.392	-34.025	-18.208	-2.392
10 - 12	-31.382	-14.473	2.436	-31.382	-14.473	2.436
10 - 1	-15.826	-3.381	9.063	-15.826	-3.381	9.063
10 - 6	-17.779	-0.870	16.039	-17.779	-0.870	16.039

SAMPLE=B2

General Linear Models Procedure

MEVH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 2	-29.407	-16.962	-4.517	-29.407	-16.962	-4.517
1 - 4	-25.742	-14.827	-3.512	-25.742	-14.827	-3.512
1 - 12	-23.537	-11.092	1.353	-23.537	-11.092	1.353
1 - 6	-9.933	2.511	14.956	-9.933	2.511	14.956
1 - 3	-18.146	2.849	23.844	-18.146	2.849	23.844
1 - 7	-7.028	2.855	12.739	-7.028	2.855	12.739
1 - 5	-4.412	2.991	10.394	-4.412	2.991	10.394
1 - 8	-9.447	2.998	15.443	-9.447	2.998	15.443
1 - 13	-17.937	3.058	24.053	-17.937	3.058	24.053
1 - 10	-9.063	3.381	15.826	-9.063	3.381	15.826
1 - 11	-8.863	3.581	16.026	-8.863	3.581	16.026
1 - 9	-8.737	3.708	16.153	-8.737	3.708	16.153
6 - 2	-36.382	-19.473	-2.564	-36.382	-19.473	-2.564
6 - 4	-33.155	-17.338	-1.522	-33.155	-17.338	-1.522
6 - 12	-30.512	-13.603	3.306	-30.512	-13.603	3.306
6 - 1	-14.956	-2.511	9.933	-14.956	-2.511	9.933
6 - 3	-23.575	0.338	24.250	-23.575	0.338	24.250
6 - 7	-14.780	0.344	15.468	-14.780	0.344	15.468
6 - 5	-13.153	0.480	14.112	-13.153	0.480	14.112
6 - 8	-16.422	0.487	17.396	-16.422	0.487	17.396
6 - 13	-23.366	0.547	24.459	-23.366	0.547	24.459
6 - 10	-16.039	0.870	17.779	-16.039	0.870	17.779
6 - 11	-15.839	1.070	17.979	-15.839	1.070	17.979
6 - 9	-15.712	1.197	18.106	-15.712	1.197	18.106
3 - 2	-43.724	-19.811	4.102	-43.724	-19.811	4.102
3 - 4	-40.829	-17.676	5.477	-40.829	-17.676	5.477
3 - 12	-37.854	-13.941	9.972	-37.854	-13.941	9.972
3 - 1	-23.844	-2.849	18.146	-23.844	-2.849	18.146
3 - 6	-24.250	-0.338	23.575	-24.250	-0.338	23.575
3 - 7	-22.679	0.006	22.692	-22.679	0.006	22.692
3 - 5	-21.578	0.142	21.862	-21.578	0.142	21.862
3 - 8	-23.764	0.149	24.062	-23.764	0.149	24.062
3 - 13	-29.078	0.209	29.496	-29.078	0.209	29.496
3 - 10	-23.380	0.532	24.445	-23.380	0.532	24.445
3 - 11	-23.180	0.732	24.645	-23.180	0.732	24.645
3 - 9	-23.054	0.859	24.772	-23.054	0.859	24.772
7 - 2	-34.941	-19.817	-4.694	-34.941	-19.817	-4.694
7 - 4	-31.574	-17.682	-3.790	-31.574	-17.682	-3.790
7 - 12	-29.071	-13.947	1.176	-29.071	-13.947	1.176
7 - 1	-12.739	-2.855	7.028	-12.739	-2.855	7.028
7 - 6	-15.468	-0.344	14.780	-15.468	-0.344	14.780
7 - 3	-22.692	-0.006	22.679	-22.692	-0.006	22.679
7 - 5	-11.207	0.136	11.478	-11.207	0.136	11.478
7 - 8	-14.981	0.143	15.266	-14.981	0.143	15.266
7 - 13	-22.483	0.203	22.888	-22.483	0.203	22.888

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
10 - 3	-24.445	-0.532	23.380	-24.445	-0.532	23.380
10 - 7	-15.650	-0.526	14.598	-15.650	-0.526	14.598
10 - 5	-14.023	-0.390	13.242	-14.023	-0.390	13.242
10 - 8	-17.292	-0.383	16.526	-17.292	-0.383	16.526
10 - 13	-24.236	-0.323	23.589	-24.236	-0.323	23.589
10 - 11	-16.709	0.200	17.109	-16.709	0.200	17.109
10 - 9	-16.582	0.327	17.236	-16.582	0.327	17.236
11 - 2	-37.452	-20.543	-3.634	-37.452	-20.543	-3.634
11 - 4	-34.225	-18.408	-2.592	-34.225	-18.408	-2.592
11 - 12	-31.582	-14.673	2.236	-31.582	-14.673	2.236
11 - 1	-16.026	-3.581	8.863	-16.026	-3.581	8.863
11 - 6	-17.979	-1.070	15.839	-17.979	-1.070	15.839
11 - 3	-24.645	-0.732	23.180	-24.645	-0.732	23.180
11 - 7	-15.850	-0.726	14.398	-15.850	-0.726	14.398
11 - 5	-14.223	-0.590	13.042	-14.223	-0.590	13.042
11 - 8	-17.492	-0.583	16.326	-17.492	-0.583	16.326
11 - 13	-24.436	-0.523	23.389	-24.436	-0.523	23.389
11 - 10	-17.109	-0.200	16.709	-17.109	-0.200	16.709
11 - 9	-16.782	0.127	17.036	-16.782	0.127	17.036
9 - 2	-37.579	-20.670	-3.761	-37.579	-20.670	-3.761
9 - 4	-34.352	-18.535	-2.718	-34.352	-18.535	-2.718
9 - 12	-31.709	-14.800	2.109	-31.709	-14.800	2.109
9 - 1	-16.153	-3.708	8.737	-16.153	-3.708	8.737
9 - 6	-18.106	-1.197	15.712	-18.106	-1.197	15.712
9 - 3	-24.772	-0.859	23.054	-24.772	-0.859	23.054
9 - 7	-15.976	-0.853	14.271	-15.976	-0.853	14.271
9 - 5	-14.349	-0.717	12.915	-14.349	-0.717	12.915
9 - 8	-17.619	-0.710	16.199	-17.619	-0.710	16.199
9 - 13	-24.563	-0.650	23.263	-24.563	-0.650	23.263
9 - 10	-17.236	-0.327	16.582	-17.236	-0.327	16.582
9 - 11	-17.036	-0.127	16.782	-17.036	-0.127	16.782

10. EXCHANGEABLE Al

units: cmol(+)/kg
mandatory for samples B1 and B2
reported by 18 laboratories

TABLE I : Exchangeable Al - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2	A1 B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5	B1 B2	1 not reported	N/R
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	B1 B2	3	R
10	B1 B2	3	N/R
11			
12			
13	A1 A2 B1 B2	3	N
14	B1 B2	5	N=R
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	2	N=R
19	A1 B1 B2	3	N=R
20	A2	1 not reported	N
21	A1 B1 B2	3	N/R
22			

TABLE II : Exchangeable Al - Summary method descriptions

method	description
1	reference, 0.1M BaCl2 1:10
2	1N KCl
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
* 6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
* 11	1M NH4Ac
12	unspecified

* not applicable

TABLE IIIa : Exchangeable A1 - descriptive statistics

sample A1 & A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
2	2	3	0.000	0.020	0.020	0.011	57.445
3	3	5	0.025	0.027	0.028	0.001	3.704
13	7	3	0.000	0.000	0.000	0.000	
21	12	3	0.020	0.020	0.020	0.000	0.000
3	3	5	0.277	0.282	0.289	0.004	1.418
13	7	3	0.000	0.000	0.000	0.000	

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	4.860	4.860	4.860	0.000	0.000
2	3	5	1.961	1.986	2.009	0.017	0.856
3	4	4	1.769	1.789	1.805	0.015	0.832
4	7	1	5.470				
6	5	3	4.480	4.527	4.570	0.045	0.996
7	9	3	1.540	1.613	1.650	0.064	3.936
9	4	3	5.050	5.350	5.570	0.269	5.029
10	7	3	4.588	4.596	4.604	0.008	0.174
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	1.020	1.103	1.210	0.097	8.803
2	3	5	0.745	0.761	0.775	0.013	1.708
3	4	4	0.468	0.482	0.509	0.019	3.963
4	7	1	2.150				
5							
6							
7	5	3	1.420	1.480	1.520	0.053	3.575
8	9	3	0.640	0.673	0.720	0.042	6.183
9							
10	4	3	0.770	0.807	0.870	0.055	6.828
11							
12							
13	7	3	1.405	1.476	1.600	0.108	7.283
14							
15							
16	4	3	1.400	1.670	1.850	0.238	14.259
17	8	3	1.510	1.560	1.610	0.050	3.205
18							
19							
20							
21	12	3	5.090	7.370	8.640	1.979	26.850
22							

TABLE IIIb : Exchangeable A1 - descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
19	3		0.23			
21	3	0.01	0.027	0.04	0.015	57.282

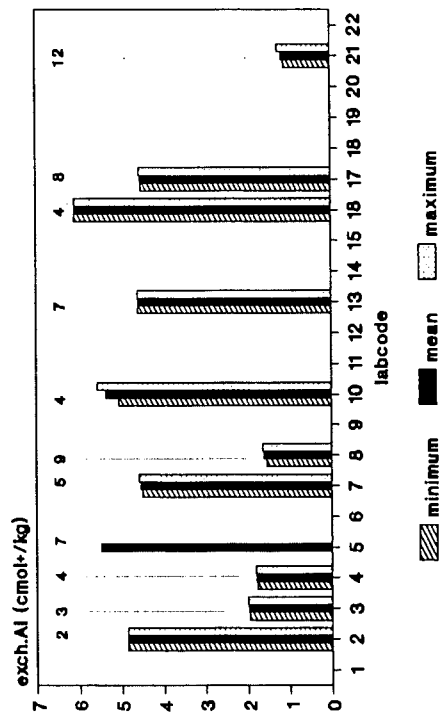
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	1.596	1.609	1.623	0.012	0.723
5	1		6.160			
6	2	3.540	3.590	3.640	0.071	1.970
7	3	3.830	3.883	3.980	0.084	2.160
8	3	1.540	1.613	1.650	0.064	3.936
9	3	5.260	5.340	5.390	0.070	1.311
10	3	3.540	3.583	3.630	0.045	1.258
11						
12						
13						
14	5	3.837	3.901	3.979	0.052	1.327
15	3		4.059			
16	3	3.780	3.867	3.980	0.103	2.654
17						
18	1		2.741			
19	3		39.000			
20	1		0.001			
21	3	3.390	3.547	3.710	0.160	4.514
22						

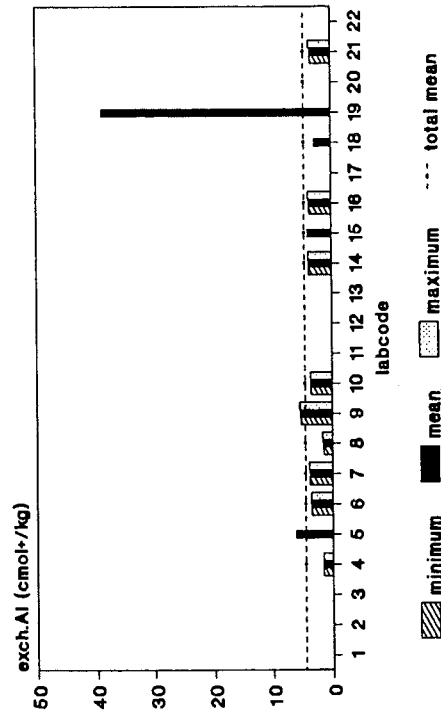
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.750	0.777	0.798	0.020	2.582
5	1		1.690			
6	2	1.310	1.380	1.450	0.099	7.174
7	3	1.600	1.620	1.630	0.017	1.069
8	3	0.640	0.673	0.720	0.042	6.183
9	3	2.950	3.090	3.180	0.123	3.977
10	3	1.980	1.993	2.010	0.015	0.766
11						
12						
13						
14	5	2.121	2.214	2.400	0.112	5.061
15	3		1.903			
16	3	1.710	1.790	1.840	0.070	3.911
17						
18	1		0.243			
19	3		19.100			
20	1		0.001			
21	3	1.190	1.787	2.270	0.549	30.719
22						

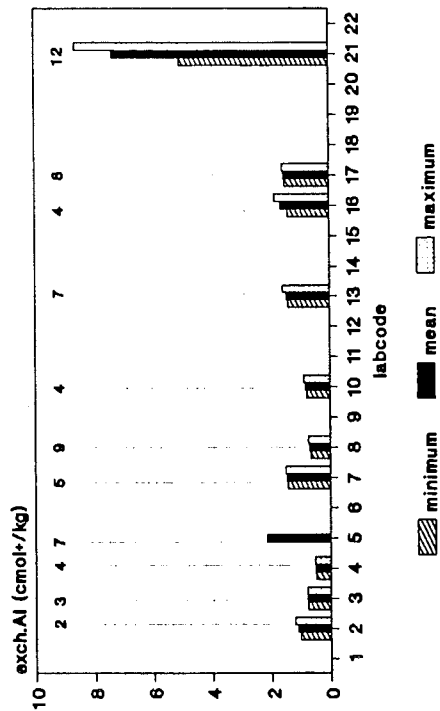
exch.AI sample B1
national methods



Exch.AI sample B1
reference method



exch.AI sample B2
national methods



Exch.AI sample B2
reference method

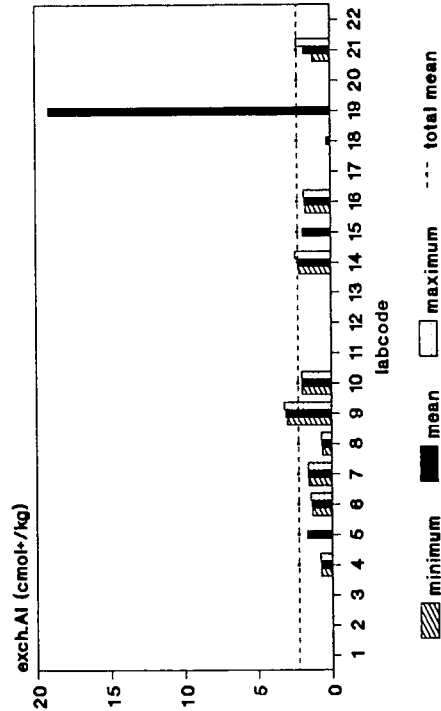


TABLE V : Exch. Al Method comparison

SAMPLE=Al

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	5	1 12 2 3 7

Number of observations in by group = 14

Dependent Variable: Exch.Al

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	0.01366669	0.97	0.4700
Error	9	0.03174167		
Corrected Total	13	0.04540836		

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	0.01366669	0.97	0.4700
Error	9	0.03174167		
Corrected Total	13	0.04540836		

Source	DF	Type I SS	F Value	Pr > F
METH	4	0.01366669	0.97	0.4700
Source	DF	Type III SS	F Value	Pr > F
METH	4	0.01366669	0.97	0.4700

TABLE IV : Exchangeable Al - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	4	0.077	0.102	0.742	0.033
A1	2	3	0.007	0.012	0.750	0.000
A1	3	1	0.027			
A1	7	3	0.000	0.000		
A1	12	3	0.020	0.000	0.000	0.000
A2	3	1	0.282			
A2	7	3	0.000	0.000		
B1	1	34	4.429	6.248	0.348	0.000
B1	2	3	4.860	0.000	0.000	0.000
B1	3	1	1.986			
B1	4	10	4.150	2.060	0.744	0.003
B1	5	3	4.527	0.045	0.996	0.877
B1	7	4	4.814	0.437	0.644	0.001
B1	8	3	4.533	0.023	0.750	0.000
B1	9	3	1.613	0.064	0.750	0.000
B1	12	3	1.167	0.081	0.848	0.235
B2	1	34	2.139	3.095	0.395	0.000
B2	2	3	1.103	0.097	0.957	0.600
B2	3	1	0.761			
B2	4	10	0.936	0.538	0.819	0.025
B2	5	3	1.480	0.053	0.893	0.363
B2	7	4	1.645	0.348	0.807	0.111
B2	8	3	1.560	0.050	1.000	0.992
B2	9	3	0.673	0.042	0.923	0.463
B2	12	3	7.370	1.979	0.805	0.126

Remark:

Labcode 3, labcode 15 and labcode 19 reported mean values for resp. 5, 3 and 3 replicates.

To calculate the total number of replicates for the method groups, SAS counts the number of reported replicates.

So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	2	3 7

Number of observations in by group = 4

Dependent Variable: Exch.Al

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	0.05964300	99999.99	0.0
Error	2	0.00000000		
Corrected Total	3	0.05964300		

R-Square	C.V.	Exch.Al Mean
1.000000	0	0.07050000

Source	DF	Type I SS	F Value	Pr > F
METH	1	0.05964300	99999.99	0.0

Source	DF	Type III SS	F Value	Pr > F
METH	1	0.05964300	99999.99	0.0

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.Al

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.Al

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 9 MSE= 0.003527
Critical Value of Studentized Range= 4.755

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 3	-0.1728	0.0505	0.0505	0.2738	
1 - 12	-0.0950	0.0575	0.0575	0.2100	
1 - 2	-0.0817	0.0708	0.0708	0.2234	
1 - 7	-0.0750	0.0775	0.0775	0.2300	
3 - 1	-0.2738	-0.0505	-0.0505	0.1728	
3 - 12	-0.2236	0.0070	0.0070	0.2376	
3 - 2	-0.2103	0.0203	0.0203	0.2509	
3 - 7	-0.2036	0.0270	0.0270	0.2576	
12 - 1	-0.2100	-0.0575	-0.0575	0.0950	
12 - 3	-0.2376	-0.0070	-0.0070	0.2236	
12 - 2	-0.1497	0.0133	0.0133	0.1764	
12 - 7	-0.1430	0.0200	0.0200	0.1830	
2 - 1	-0.2234	-0.0708	-0.0708	0.0817	
2 - 3	-0.2509	-0.0203	-0.0203	0.2103	
2 - 12	-0.1764	-0.0133	-0.0133	0.1497	
2 - 7	-0.1564	0.0067	0.0067	0.1697	
7 - 1	-0.2300	-0.0775	-0.0775	0.0750	
7 - 3	-0.2576	-0.0270	-0.0270	0.2036	
7 - 12	-0.1830	-0.0200	-0.0200	0.1430	
7 - 2	-0.1697	-0.0067	-0.0067	0.1564	

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.A1

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 55 MSE= 24.12458

Critical Value of Studentized Range= 4.566

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Limit	Confidence Limit	Between Means	Upper Confidence Limit	Upper Limit
2 - 7	-12.065	-12.065	0.046	12.156	12.156
2 - 8	-12.620	-12.620	0.327	13.274	13.274
2 - 5	-12.614	-12.614	0.333	13.280	13.280
2 - 1	-9.119	-9.119	0.431	9.981	9.981
2 - 4	-9.729	-9.729	0.710	11.148	11.148
2 - 3	-15.436	-15.436	2.874	21.184	21.184
2 - 9	-9.700	-9.700	3.247	16.194	16.194
2 - 12	-9.254	-9.254	3.693	16.640	16.640
7 - 2	-12.156	-12.156	-0.046	12.065	12.065
7 - 8	-11.830	-11.830	0.281	12.392	12.392
7 - 5	-11.823	-11.823	0.288	12.399	12.399
7 - 1	-7.996	-7.996	0.386	8.767	8.767
7 - 4	-8.717	-8.717	0.664	10.045	10.045
7 - 3	-14.900	-14.900	2.829	20.557	20.557
7 - 9	-8.910	-8.910	3.201	15.312	15.312
7 - 12	-8.463	-8.463	3.648	15.759	15.759
8 - 2	-13.274	-13.274	-0.327	12.620	12.620
8 - 7	-12.392	-12.392	-0.281	11.830	11.830
8 - 5	-12.940	-12.940	0.007	12.954	12.954
8 - 1	-9.446	-9.446	0.104	9.655	9.655
8 - 4	-10.055	-10.055	0.383	10.821	10.821
8 - 3	-15.762	-15.762	2.547	20.857	20.857
8 - 9	-10.027	-10.027	2.920	15.867	15.867
8 - 12	-9.580	-9.580	3.367	16.314	16.314
5 - 2	-13.280	-13.280	-0.333	12.614	12.614
5 - 7	-12.399	-12.399	-0.288	11.823	11.823
5 - 8	-12.954	-12.954	-0.007	12.940	12.940
5 - 1	-9.452	-9.452	0.098	9.648	9.648
5 - 4	-10.062	-10.062	0.376	10.814	10.814
5 - 3	-15.769	-15.769	2.541	20.850	20.850
5 - 9	-10.034	-10.034	2.913	15.860	15.860
5 - 12	-9.587	-9.587	3.360	16.307	16.307
1 - 2	-9.981	-9.981	-0.431	9.119	9.119
1 - 7	-8.767	-8.767	-0.386	7.996	7.996
1 - 8	-9.655	-9.655	-0.104	9.446	9.446

SAMPLE=B1

General Linear Models Procedure

Class Level Information

Class Levels Values

METH 9 1 12 2 3 4 5 7 8 9

Number of observations in by group = 64

Dependent Variable: Exch.A1

Source	DF	Sum of Squares	F Value	Pr > F
Model	8	57.40300101	0.30	0.9638
Error	55	1326.85177443		
Corrected Total	63	1384.25477544		
R-Square		C.V.		Exch.A1 Mean
0.041469		119.3286		4.11609375
Source	DF	Type I SS	F Value	Pr > F
METH	8	57.40300101	0.30	0.9638
Source	DF	Type III SS	F Value	Pr > F
METH	8	57.40300101	0.30	0.9638

SAMPLE=B2

SAMPLE=B1

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Class	Levels	Values
METH	9	1 12 2 3 4 5 7 8 9

Number of observations in by group = 64

Dependent Variable: Exch.Al

Source	DF	Sum of Squares	F Value	Pr > F
Model	8	109.55270461	2.30	0.0331
Error	55	327.01519114		
Corrected Total	63	436.56789575		

R-Square C.V. Exch.Al Mean

0.250941 123.8585 1.96868750

Source	DF	Type I SS	F Value	Pr > F
METH	8	109.55270461	2.30	0.0331
Source	DF	Type III SS	F Value	Pr > F
METH	8	109.55270461	2.30	0.0331

Simultaneous

METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit

1 - 5	-9.648	-0.098	9.452
1 - 4	-5.426	0.278	5.983
1 - 3	-13.645	2.443	18.531
1 - 9	-6.735	2.816	12.366
1 - 12	-6.288	3.262	12.812

4 - 2	-11.148	-0.710	9.729
4 - 7	-10.045	-0.664	8.717
4 - 8	-10.821	-0.383	10.055
4 - 5	-10.814	-0.376	10.062
4 - 1	-5.983	-0.278	5.426

4 - 3	-14.466	2.165	18.795
4 - 9	-7.901	2.537	12.975
4 - 12	-7.454	2.984	13.422

3 - 2	-21.184	-2.874	15.436
3 - 7	-20.557	-2.829	14.900
3 - 8	-20.857	-2.547	15.762
3 - 5	-20.850	-2.541	15.769
3 - 1	-18.531	-2.443	13.645

3 - 4	-18.795	-2.165	14.466
3 - 9	-17.937	0.373	18.682
3 - 12	-17.490	0.819	19.129

9 - 2	-16.194	-3.247	9.700
9 - 7	-15.312	-3.201	8.910
9 - 8	-15.867	-2.920	10.027
9 - 5	-15.860	-2.913	10.034
9 - 1	-12.366	-2.816	6.735

9 - 4	-12.975	-2.537	7.901
9 - 3	-18.682	-0.373	17.937
9 - 12	-12.500	0.447	13.394

12 - 2	-16.640	-3.693	9.254
12 - 7	-15.759	-3.648	8.463
12 - 8	-16.314	-3.367	9.580
12 - 5	-16.307	-3.360	9.587
12 - 1	-12.812	-3.262	6.288

12 - 4	-13.422	-2.984	7.454
12 - 3	-19.129	-0.819	17.490
12 - 9	-13.394	-0.447	12.500

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.A1

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 55 MSE= 5.945731
Critical Value of Studentized Range= 4.566

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous Lower Confidence Limit
Simultaneous Upper Confidence Limit
Difference Between Means
METH Comparison

Simultaneous Lower Confidence Limit
Simultaneous Upper Confidence Limit
Difference Between Means
METH Comparison

12	- 1	0.490	5.231	9.972	***	5	- 8	-6.507	-0.080	6.347	
12	- 7	-0.287	5.725	11.738		5	- 2	-6.051	0.377	6.804	
12	- 8	-0.617	5.810	12.237		5	- 4	-4.638	0.544	5.726	
12	- 5	-0.537	5.890	12.317		5	- 3	-8.371	0.719	9.809	
12	- 2	-0.161	6.267	12.694		5	- 9	-5.621	0.807	7.234	
12	- 4	1.252	6.434	11.616	***	2	- 12	-12.694	-6.267	0.161	
12	- 3	-2.481	6.609	15.899	***	2	- 1	-5.777	-1.036	3.705	
12	- 9	0.269	6.697	13.124	***	2	- 7	-6.554	-0.541	5.471	
1	- 12	-9.972	-5.231	-0.490	***	2	- 8	-6.884	-0.457	5.971	
1	- 7	-3.667	0.495	4.656		2	- 5	-6.804	-0.377	6.051	
1	- 8	-4.162	0.579	5.320		2	- 4	-5.015	0.167	5.349	
1	- 5	-4.082	0.659	5.400		2	- 3	-8.747	0.342	9.432	
1	- 2	-3.705	1.036	5.777		2	- 9	-5.997	0.430	6.857	
1	- 4	-1.629	1.203	4.035		4	- 12	-11.616	-6.434	-1.252	***
1	- 3	-6.609	1.378	9.365	***	4	- 1	-4.035	-1.203	1.629	
1	- 9	-3.275	1.466	6.207	***	4	- 7	-5.366	-0.709	3.948	
7	- 12	-11.738	-5.725	0.287		4	- 8	-5.806	-0.624	4.558	
7	- 1	-4.656	-0.495	3.667		4	- 5	-5.726	-0.544	4.638	
7	- 8	-5.928	0.085	6.097		4	- 2	-5.349	-0.167	5.015	
7	- 5	-5.848	0.165	6.177		4	- 3	-8.081	0.175	8.431	
7	- 2	-5.471	0.541	6.554		4	- 9	-4.919	0.263	5.445	
7	- 4	-3.948	0.709	5.366		3	- 12	-15.699	-6.609	2.481	
7	- 3	-7.917	0.884	9.685		3	- 1	-9.365	-1.378	6.609	
7	- 9	-5.041	0.971	6.984		3	- 7	-9.685	-0.884	7.917	
8	- 12	-12.237	-5.810	0.617		3	- 8	-9.889	-0.799	8.291	
8	- 1	-5.320	-0.579	4.162		3	- 5	-9.809	-0.719	8.371	
8	- 7	-6.097	-0.085	5.928		3	- 2	-9.432	-0.342	8.747	
8	- 5	-6.347	0.080	6.507		3	- 4	-8.431	-0.175	8.081	
8	- 2	-5.971	0.457	6.884		3	- 9	-9.002	0.088	9.177	
8	- 4	-4.558	0.624	5.806		9	- 12	-13.124	-6.697	-0.269	***
8	- 3	-8.291	0.799	9.889		9	- 1	-6.207	-1.466	3.275	
8	- 9	-5.541	0.887	7.314		9	- 7	-6.984	-0.971	5.041	
5	- 12	-12.317	-5.890	0.537		9	- 8	-7.314	-0.887	5.541	
5	- 1	-5.400	-0.659	4.082		9	- 5	-7.234	-0.807	5.621	
5	- 7	-6.177	-0.165	5.848		9	- 2	-6.857	-0.430	5.997	
						9	- 4	-5.445	-0.263	4.919	
						9	- 3	-9.177	-0.088	9.002	

11. EXCHANGEABLE Fe

units: cmol+/kg
mandatory for B1 and B2
reported by 14 laboratories

TABLE I : Exchangeable Fe - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2			
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5			
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	B1 B2	3	R
10	B1 B2	3	N/R
11			
12			
13	A1 A2 B1 B2	3	N
14	A1 A2 B1 B2	5	N=R
15			
16	B1 B2	3	N/R B1: R
17			
18			
19	A1 B1 B2	3	N=R
20	B1 B2	1 not reported	N
21	A1 B1 B2	3	R
22	B1 B2	3	R

TABLE II : Exchangeable Fe - Summary method description

method	description
1	reference, 0.1M BaCl2 1:10
2	1N KCl
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, next: 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
11	1M NH4Ac
12	unspecified

TABLE IIIa : Exchangeable Fe - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
3	A1	3	5	0.001	0.001	0.001	0.000	0.000
13	A1	7	3	0.000	0.000	0.000	0.000	0.000
3	A2	3	5	0.031	0.032	0.033	0.001	31.250
13	A2	7	3	0.042	0.045	0.048	0.003	6.840

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.001	0.002	0.003	0.000	0.000
4	4	4	0.003	0.004	0.007	0.002	50.000
5							
6							
7	5	3	0.004	0.005	0.006	0.001	24.744
8	9	3	0.15	0.150	0.150	0.000	0.000
9							
10	4	3	0.01	0.010	0.010	0.000	0.000
11							
12							
13	7	3	0.031	0.032	0.033	0.001	3.646
14							
15							
16	4	3	0	0.000	0.000	0.000	
17							
18							
19							
20	10	1		0.043			
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.08	0.083	0.085	0.002	2.410
4	4	4	0.098	0.101	0.107	0.004	4.042
5							
6							
7	5	3	0.068	0.070	0.073	0.003	3.578
8	9	3	0.01	0.013	0.020	0.006	43.301
9							
10	4	3	0.04	0.047	0.050	0.006	12.372
11							
12							
13	7	3	0.557	0.593	0.663	0.060	10.172
14							
15							
16	4	3	0.123	0.145	0.165	0.021	14.538
17							
18							
19							
20	10	1		0.025			
21							
22							

TABLE IIIb : Exchangeable Fe - descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
14	2	0.001	0.002	0.002	0.001	47.140
19	3		0.150			
21	3	0.010	0.040	0.080	0.036	90.139

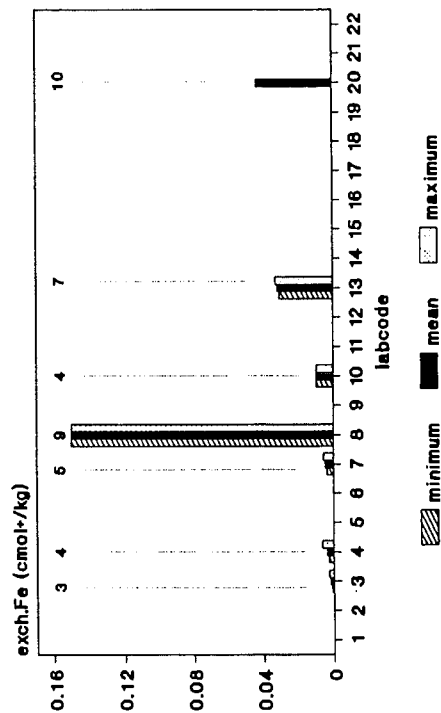
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	3	0.000	0.000	0.000	0.000	
5						
6	2	0.000	0.000	0.000	0.000	
7	3	0.003	0.004	0.004	0.001	15.746
8	3	0.030	0.030	0.030	0.000	0.000
9	3	0.002	0.002	0.002	0.000	0.000
10	3	0.010	0.010	0.010	0.000	0.000
11						
12						
13						
14	5	0.002	0.004	0.010	0.003	83.163
15						
16	3	0.009	0.012	0.015	0.003	24.771
17						
18						
19	3		0.200			
20						
21	3	0.010	0.023	0.040	0.015	65.465
22	3	0.001	0.002	0.004	0.002	65.465

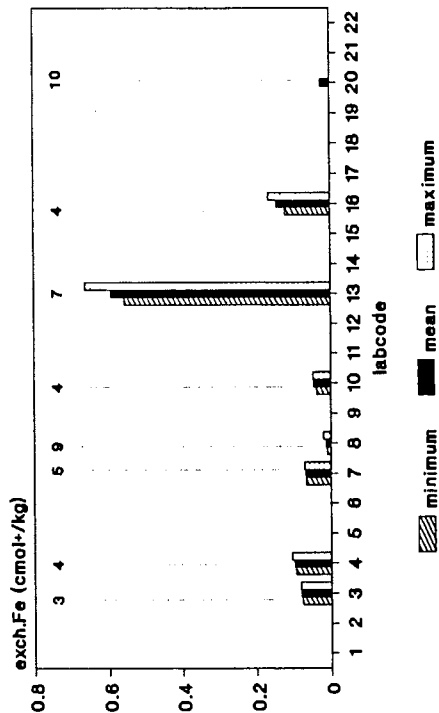
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.197	0.206	0.214	0.008	3.654
5						
6	2	0.070	0.070	0.070	0.000	0.000
7	3	0.086	0.089	0.091	0.003	2.838
8	3	0.004	0.005	0.005	0.001	12.372
9	3	0.274	0.283	0.290	0.008	2.939
10	3	0.200	0.210	0.220	0.010	4.762
11						
12						
13						
14	5	0.211	0.231	0.255	0.016	6.851
15						
16	3	0.148	0.153	0.158	0.005	3.283
17						
18						
19	3		1.700			
20						
21	3	0.200	0.217	0.230	0.015	7.050
22	3	0.066	0.068	0.070	0.002	2.941

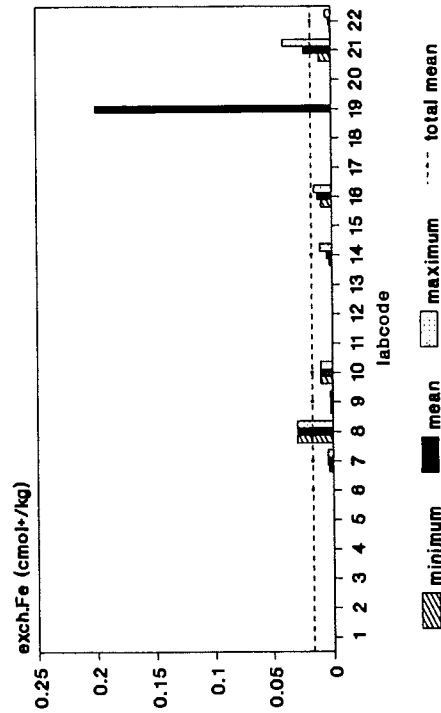
exch.Fe sample B1
national methods



exch.Fe sample B2
national methods



exch.Fe sample B1
reference method



exch.Fe sample B2
reference method

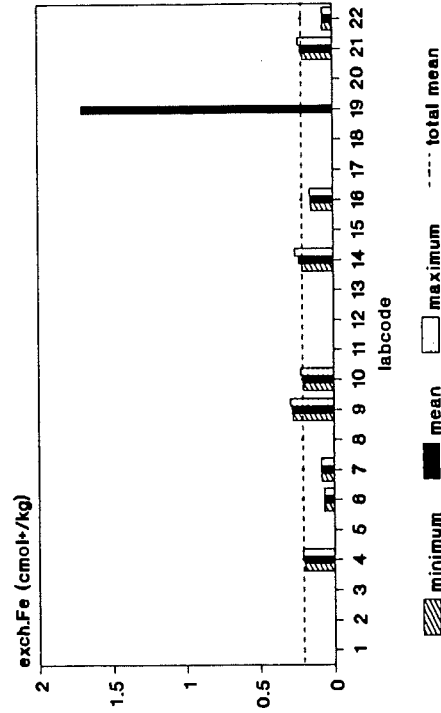


TABLE V : Exch.Fe Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	3	1 3 7

Number of observations in by group = 13

Dependent Variable: Exch.Fe

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	0.00250692	0.58	0.5778
Error	10	0.02162400		
Corrected Total	12	0.02413092		
R-Square				
C.V.			Exch.Fe Mean	
0.103888			220.6281	0.02107692

Source	DF	Type I SS	F Value	Pr > F
METH	2	0.00250692	0.58	0.5778
Source	DF	Type III SS	F Value	Pr > F
METH	2	0.00250692	0.58	0.5778

Tukey's Studentized Range (HSD) Test for variable: Exch.Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 10 MSE= 0.002162
Critical Value of Studentized Range= 3.877

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 3	-0.1050	0.0293	0.1637
1 - 7	-0.0546	0.0303	0.1153
3 - 1	-0.1637	-0.0293	0.1050
3 - 7	-0.1462	0.0010	0.1482
7 - 1	-0.1153	-0.0303	0.0546
7 - 3	-0.1482	-0.0010	0.1462

TABLE IV : Exchangeable Fe - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	0.030	0.052	0.676	0.001
A1	3	1	0.001			
A1	7	3	0.000	0.000		
A2	3	1	0.032			
A2	7	3	0.045	0.003	0.964	0.637
B1	1	33	0.014	0.035	0.397	0.000
B1	3	1	0.002			
B1	4	10	0.005	0.004	0.833	0.036
B1	5	3	0.005	0.001	0.750	0.000
B1	7	3	0.032	0.001	0.750	0.000
B1	9	3	0.150	0.000	0.000	0.000
B1	10	1	0.043			
B2	1	33	0.209	0.281	0.442	0.000
B2	3	1	0.083			
B2	4	10	0.098	0.041	0.934	0.474
B2	5	3	0.070	0.003	0.987	0.780
B2	7	3	0.593	0.060	0.771	0.047
B2	9	3	0.013	0.006	0.750	0.000
B2	10	1	0.025			

Remark:

Labcode 3 and labcode 19 reported mean values for resp. 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" value in this table for methods 3 and 1 are different from the real number of replicates.

Exch.Fe Method comparison

Exch.Fe Method comparison

SAMPLE=A2

SAMPLE=B1

General Linear Models Procedure
Class Level InformationGeneral Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	2	3 7

Class	Levels	Values
METH	7	1 10 3 4 5 7 9

Number of observations in by group = 4

Number of observations in by group = 54

Dependent Variable: Exch.Fe

Dependent Variable: Exch.Fe

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	0.00012033	12.89	0.0696
Error	2	0.00001867		
Corrected Total	3	0.00013900		
R-Square				
C.V.		7.361567		
Exch.Fe Mean				0.04150000

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.05607663	11.20	0.0001
Error	47	0.03920470		
Corrected Total	53	0.09528133		
R-Square				
C.V.		139.0020		
Exch.Fe Mean				0.02077778

Source	DF	Type I SS	F Value	Pr > F
METH	1	0.00012033	12.89	0.0696
Source	DF	Type III SS	F Value	Pr > F
METH	1	0.00012033	12.89	0.0696

Source	DF	Type I SS	F Value	Pr > F
METH	6	0.05607663	11.20	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.05607663	11.20	0.0001

Tukey's Studentized Range (HSD) Test for variable: Exch.Fe

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of 2 means with 2 error degrees of freedom failed.

Exch.Fe Method comparison

Exch.Fe Method comparison

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 47 MSE= 0.000834

Critical Value of Studentized Range= 4.355

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
9 - 10	0.00430	0.10700	0.20970	-0.05861	-0.00007	0.05848
9 - 7	0.04571	0.11833	0.19095	-0.09068	0.00260	0.09588
9 - 1	0.08206	0.13570	0.18933	-0.25070	-0.14800	-0.04530
9 - 5	0.07271	0.14533	0.21795	-0.16578	-0.04100	0.08478
9 - 4	0.08685	0.14540	0.20395	-0.13237	-0.02967	0.07303
9 - 3	0.04530	0.14800	0.25070	-0.10258	-0.01230	0.07798
10 - 9	-0.20970	-0.10700	-0.00430	-0.10537	-0.00267	0.10003
10 - 7	-0.09137	0.01133	0.11403	-0.09588	-0.00260	0.09068
10 - 1	-0.06158	0.02870	0.11898			
10 - 5	-0.06437	0.03833	0.14103			
10 - 4	-0.05488	0.03840	0.13168			
10 - 3	-0.08478	0.04100	0.16678			
7 - 9	-0.19095	-0.11833	-0.04571			
7 - 10	-0.11403	-0.01133	0.09137			
7 - 1	-0.03627	0.01736	0.07100			
7 - 5	-0.04562	0.02700	0.09962			
7 - 4	-0.03148	0.02707	0.08561			
7 - 3	-0.07303	0.02967	0.13237			
1 - 9	-0.18933	-0.13570	-0.08206			
1 - 10	-0.11898	-0.02870	0.06158			
1 - 7	-0.07100	-0.01736	0.03627			
1 - 5	-0.04400	0.00964	0.06327			
1 - 4	-0.02240	0.00970	0.04181			
1 - 3	-0.07798	0.01230	0.10258			
5 - 9	-0.21795	-0.14533	-0.07271			
5 - 10	-0.14103	-0.03833	0.06437			
5 - 7	-0.09962	-0.02700	0.04562			
5 - 1	-0.06327	-0.00964	0.04400			
5 - 4	-0.05848	0.00007	0.05861			
5 - 3	-0.10003	0.00267	0.10537			
4 - 9	-0.20395	-0.14540	-0.08685			
4 - 10	-0.13168	-0.03840	0.05488			
4 - 7	-0.08561	-0.02707	0.03148			
4 - 1	-0.04181	-0.00970	0.02240			

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Exch.Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 47 MSE= 0.054118
Critical Value of Studentized Range= 4.355

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7	1	-0.0476	0.3844	0.3844	0.8164	0.9671
7	4	0.0239	0.4955	0.4955	0.9671	0.9671
7	3	-0.3169	0.5103	0.5103	1.3376	1.3376
7	5	-0.0619	0.5230	0.5230	1.1079	1.1079
7	10	-0.2589	0.5683	0.5683	1.3956	1.3956
7	9	-0.0049	0.5800	0.5800	1.1649	1.1649
1	7	-0.8164	-0.3844	-0.3844	0.0476	0.0476
1	4	-0.1475	0.1111	0.1111	0.3697	0.3697
1	3	-0.6012	0.1259	0.1259	0.8531	0.8531
1	5	-0.2934	0.1386	0.1386	0.5706	0.5706
1	10	-0.5432	0.1839	0.1839	0.9111	0.9111
1	9	-0.2364	0.1956	0.1956	0.6276	0.6276
4	7	-0.9671	-0.4955	-0.4955	-0.0239	-0.0239
4	1	-0.3697	-0.1111	-0.1111	0.1475	0.1475
4	3	-0.7366	0.0148	0.0148	0.7662	0.7662
4	5	-0.4441	0.0275	0.0275	0.4991	0.4991
4	10	-0.6786	0.0728	0.0728	0.8242	0.8242
4	9	-0.3871	0.0845	0.0845	0.5561	0.5561
3	7	-1.3376	-0.5103	-0.5103	0.3169	0.3169
3	1	-0.8531	-0.1259	-0.1259	0.6012	0.6012
3	4	-0.7662	-0.0148	-0.0148	0.7366	0.7366
3	5	-0.8146	0.0127	0.0127	0.8399	0.8399
3	10	-0.9551	0.0580	0.0580	1.0711	1.0711
3	9	-0.7576	0.0697	0.0697	0.8969	0.8969
5	7	-1.1079	-0.5230	-0.5230	0.0619	0.0619
5	1	-0.5706	-0.1386	-0.1386	0.2934	0.2934
5	4	-0.4991	-0.0275	-0.0275	0.4441	0.4441
5	3	-0.8399	-0.0127	-0.0127	0.8146	0.8146
5	10	-0.7819	0.0453	0.0453	0.8726	0.8726
5	9	-0.5279	0.0570	0.0570	0.6419	0.6419
10	7	-1.3956	-0.5683	-0.5683	0.2589	0.2589
10	1	-0.9111	-0.1839	-0.1839	0.5432	0.5432
10	4	-0.8242	-0.0728	-0.0728	0.6786	0.6786
10	3	-1.0711	-0.0580	-0.0580	0.9551	0.9551

SAMPLE=B2

General Linear Models Procedure

Class Level Information

Class Levels Values

METH 7 1 10 3 4 5 7 9

Number of observations in by group = 54

Dependent Variable: Exch.Fe

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.75900756	2.34	0.0467
Error	47	2.54355148		
Corrected Total	53	3.30255904		
R-Square		C.V.		Exch.Fe Mean
	0.229824	125.4713		0.18540741
Source	DF	Type I SS	F Value	Pr > F
METH	6	0.75900756	2.34	0.0467
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.75900756	2.34	0.0467

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
10 - 5	-0.8726	-0.0453	0.7819	
10 - 9	-0.8156	0.0117	0.8389	
9 - 7	-1.1649	-0.5800	0.0049	
9 - 1	-0.6276	-0.1956	0.2364	
9 - 4	-0.5561	-0.0845	0.3871	
9 - 3	-0.8969	-0.0697	0.7576	
9 - 5	-0.6419	-0.0570	0.5279	
9 - 10	-0.8389	-0.0117	0.8156	

12. EXCHANGEABLE Mn

units: cmol+/kg
mandatory for samples B1 and B2
reported by 14 laboratories

TABLE II : Exchangeable Mn - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2			
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5			
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	B1 B2	3	R
10	B1 B2	3	N/R
11			
12			
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N
15			
16	B1 B2	3	N/R
17	B1 B2	3	N
18			
19	A1 B1 B2	3	N=R
20			
21	A1 B1 B2	3	R
22	B1 B2	3	R

TABLE I : Exchangeable Mn - Summary method description

method	description
1	reference
*	1N KCl
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
*	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, next: 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
*	10 0.1M BaCl2
*	11 1M NH4Ac
*	12 unspecified

* not applicable

TABLE IIIa : Exchangeable Mn - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
3	A1	3	5		0.013			
13	A1	7	3	0.011	0.011	0.011	0.000	0.000
3	A2	3	5	1.058	1.076	1.088	0.012	1.115
13	A2	7	3	1.433	1.462	1.504	0.037	2.547

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.044	0.045	0.045	0.000	0.000
4	4	4	0.080	0.082	0.083	0.001	1.725
5							
6							
7	5	3	0.081	0.081	0.082	0.001	0.710
8	9	3	0.020	0.027	0.030	0.006	21.651
9							
10	4	3	0.060	0.073	0.090	0.015	20.830
11							
12							
13	7	3	0.097	0.098	0.099	0.001	1.174
14							
15							
16	4	3	0.084	0.087	0.090	0.003	3.498
17	8	3	0.017	0.017	0.017	0.000	0.000
18							
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.009	0.010	0.010	0.000	0.000
4	4	4	0.013	0.014	0.015	0.001	6.719
5							
6							
7	5	3	0.012	0.013	0.013	0.001	4.558
8	9	3	0.030	0.030	0.030	0.000	0.000
9							
10	4	3	0.010	0.010	0.010	0.000	0.000
11							
12							
13	7	3	0.016	0.017	0.019	0.002	10.189
14							
15							
16	4	3	0.009	0.011	0.012	0.002	15.746
17	8	3	0.002	0.003	0.003	0.001	21.651
18							
19							
20							
21							
22							

TABLE IIIb : Exchangeable Mn - descriptive statistics

sample A1 - reference method

labcode	sample	repl	minimum	mean	maximum	std	cv
14	A1	5	0.006	0.006	0.006	0.000	0.000
19	A1	3		0.050			
21	A1	3	0.010	0.010	0.010	0.000	0.000

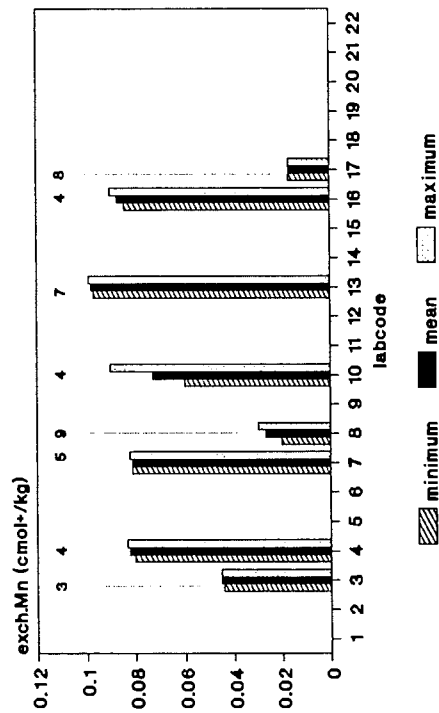
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.082	0.084	0.085	0.002	1.791
5						
6	2	0.080	0.080	0.080	0.000	0.000
7	3	0.082	0.082	0.083	0.001	0.701
8	3	0.040	0.040	0.040	0.000	0.000
9	3	0.083	0.084	0.086	0.002	1.811
10	3	0.080	0.080	0.080	0.000	0.000
11						
12						
13						
14	5	0.080	0.081	0.083	0.001	1.401
15						
16	3	0.079	0.081	0.082	0.002	1.894
17						
18						
19	3		0.800			
20						
21	3	0.100	0.103	0.110	0.006	5.587
22	3	0.066	0.068	0.070	0.002	2.941

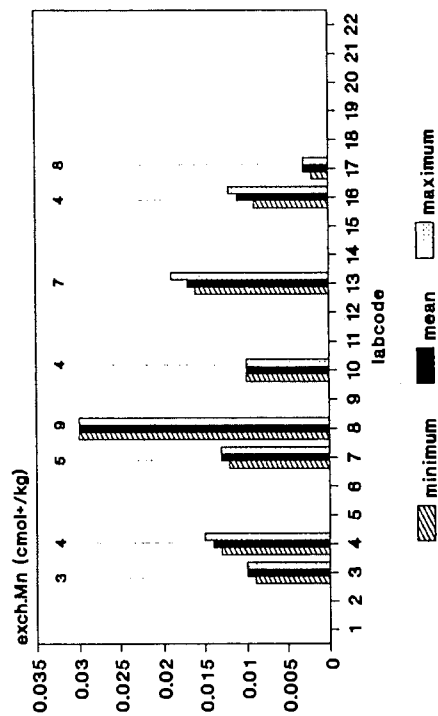
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.017	0.018	0.019	0.001	5.246
5						
6	2	0.010	0.010	0.010	0.000	0.000
7	3	0.015	0.016	0.016	0.001	3.685
8	3	0.006	0.006	0.007	0.001	9.116
9	3	0.017	0.017	0.018	0.001	3.331
10	3	0.020	0.020	0.020	0.000	0.000
11						
12						
13						
14	5	0.018	0.019	0.021	0.001	7.213
15						
16	3	0.017	0.018	0.019	0.001	5.556
17						
18						
19	3		0.200			
20						
21	3	0.030	0.030	0.030	0.000	0.000
22	3	0.012	0.013	0.014	0.001	9.116

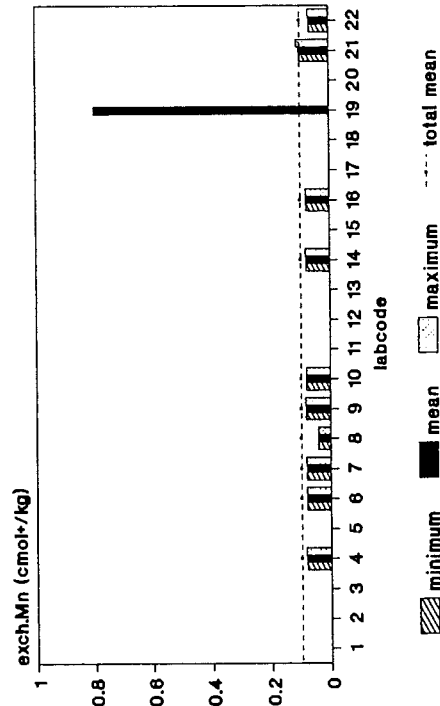
exch.Mn sample B1
national methods



exch.Mn sample B2
national methods



exch.Mn sample B1
reference method



exch.Mn sample B2
reference method

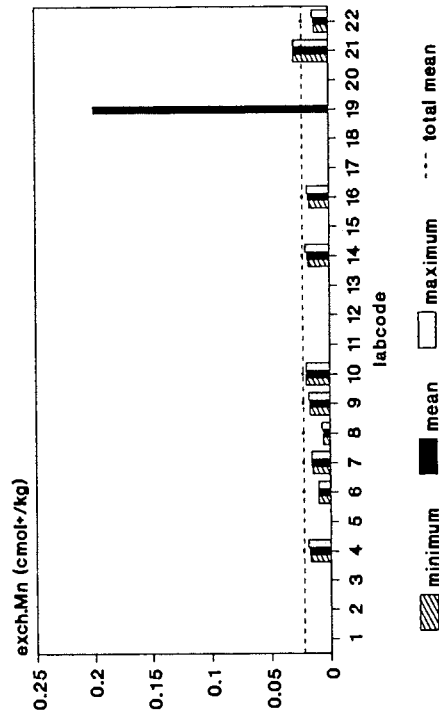


TABLE V : exch.Mn Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 3 1 3 7

Number of observations in by group = 13

Dependent Variable: EXCH.Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	0.00000444	0.01	0.9865
Error	10	0.00163556		
Corrected Total	12	0.00164000		
R-Square		C.V.		EXCH.Mn Mean
	0.002710	106.5740		0.01200000

Source	DF	Type I SS	F Value	Pr > F
METH	2	0.00000444	0.01	0.9865
Source	DF	Type III SS	F Value	Pr > F
METH	2	0.00000444	0.01	0.9865

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 10 MSE= 0.000164

Critical Value of Studentized Range= 3.877

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous		
		Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
3 - 1		-0.03618	0.00078	0.03773
3 - 7		-0.03848	0.00200	0.04248
1 - 3		-0.03773	-0.00078	0.03618
1 - 7		-0.02215	0.00122	0.02459
7 - 3		-0.04248	-0.00200	0.03848
7 - 1		-0.02459	-0.00122	0.02215

TABLE IV : Exchangeable Mn - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	0.012	0.014	0.488	0.000
A1	3	1	0.013			
A1	7	3	0.011	0.000	0.000	0.000
A2	3	1	1.076			
A2	7	3	1.462	0.037	0.909	0.413
B1	1	33	0.101	0.126	0.263	0.000
B1	3	1	0.045			
B1	4	10	0.081	0.009	0.836	0.039
B1	5	3	0.081	0.001	0.750	0.000
B1	7	3	0.098	0.001	0.750	0.000
B1	8	3	0.017	0.000		
B1	9	3	0.027	0.006	0.750	0.000
B2	1	33	0.023	0.032	0.322	0.000
B2	3	1	0.010			
B2	4	10	0.012	0.002	0.910	0.271
B2	5	3	0.013	0.001	0.750	0.000
B2	7	3	0.017	0.002	0.750	0.000
B2	8	3	0.003	0.001	0.750	0.000
B2	9	3	0.030	0.000		

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 3 and 5 replicates.

To calculate the total number of replicates for the method-groups, SAS counts the number of reported results.

So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 3 4 5 7 8 9

Number of observations in by group = 56

Dependent Variable: EXCH.Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.03426580	0.55	0.7705
Error	49	0.51243418		
Corrected Total	55	0.54669998		
R-Square		C.V.		EXCH.Mn Mean
		118.2482		0.08648214

Source	DF	Type I SS	F Value	Pr > F
METH	6	0.03426580	0.55	0.7705
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.03426580	0.55	0.7705

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mn

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	2	3 7

Number of observations in by group = 4

Dependent Variable: EXCH.Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	0.11174700	80.57	0.0122
Error	2	0.00277400		
Corrected Total	3	0.11452100		
R-Square		C.V.		EXCH.Mn Mean
		2.727385		1.36550000

Source	DF	Type I SS	F Value	Pr > F
METH	1	0.11174700	80.57	0.0122
Source	DF	Type III SS	F Value	Pr > F
METH	1	0.11174700	80.57	0.0122

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
9 - 3	-0.3813	0.3447	-0.0183	-0.3813	0.3447
9 - 8	-0.2470	0.2663	0.0097	-0.2470	0.2663
8 - 1	-0.2731	0.1060	-0.0835	-0.2731	0.1060
8 - 7	-0.3380	0.1753	-0.0813	-0.3380	0.1753
8 - 5	-0.3210	0.1923	-0.0643	-0.3210	0.1923
8 - 4	-0.2709	0.1429	-0.0640	-0.2709	0.1429
8 - 3	-0.3910	0.3350	-0.0280	-0.3910	0.3350
8 - 9	-0.2663	0.2470	-0.0097	-0.2663	0.2470

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH. Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 49 MSE= 0.010458

Critical Value of Studentized Range= 4.347

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 7	-0.1874	0.0022	0.0022	0.1918	0.1918
1 - 5	-0.1704	0.0192	0.0192	0.2088	0.2088
1 - 4	-0.0939	0.0195	0.0195	0.1330	0.1330
1 - 3	-0.2635	0.0555	0.0555	0.3746	0.3746
1 - 9	-0.1157	0.0739	0.0739	0.2634	0.2634
1 - 8	-0.1060	0.0835	0.0835	0.2731	0.2731
7 - 1	-0.1918	-0.0022	-0.0022	0.1874	0.1874
7 - 5	-0.2397	0.0170	0.0170	0.2737	0.2737
7 - 4	-0.1896	0.0173	0.0173	0.2243	0.2243
7 - 3	-0.3097	0.0533	0.0533	0.4163	0.4163
7 - 9	-0.1850	0.0717	0.0717	0.3283	0.3283
7 - 8	-0.1753	0.0813	0.0813	0.3380	0.3380
5 - 1	-0.2088	-0.0192	-0.0192	0.1704	0.1704
5 - 7	-0.2737	-0.0170	-0.0170	0.2397	0.2397
5 - 4	-0.2066	0.0003	0.0003	0.2073	0.2073
5 - 3	-0.3267	0.0363	0.0363	0.3993	0.3993
5 - 9	-0.2020	0.0547	0.0547	0.3113	0.3113
5 - 8	-0.1923	0.0643	0.0643	0.3210	0.3210
4 - 1	-0.1330	-0.0195	-0.0195	0.0939	0.0939
4 - 7	-0.2243	-0.0173	-0.0173	0.1896	0.1896
4 - 5	-0.2073	-0.0003	-0.0003	0.2066	0.2066
4 - 3	-0.2937	0.0360	0.0360	0.3657	0.3657
4 - 9	-0.1526	0.0543	0.0543	0.2613	0.2613
4 - 8	-0.1429	0.0640	0.0640	0.2709	0.2709
3 - 1	-0.3746	-0.0555	-0.0555	0.2635	0.2635
3 - 7	-0.4163	-0.0533	-0.0533	0.3097	0.3097
3 - 5	-0.3993	-0.0363	-0.0363	0.3267	0.3267
3 - 4	-0.3657	-0.0360	-0.0360	0.2937	0.2937
3 - 9	-0.3447	0.0183	0.0183	0.3813	0.3813
3 - 8	-0.3350	0.0280	0.0280	0.3910	0.3910
9 - 1	-0.2634	-0.0739	-0.0739	0.1157	0.1157
9 - 7	-0.3283	-0.0717	-0.0717	0.1850	0.1850
9 - 5	-0.3113	-0.0547	-0.0547	0.2020	0.2020
9 - 4	-0.2613	-0.0543	-0.0543	0.1526	0.1526

----- SAMPLE=B2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 3 4 5 7 8 9

Number of observations in by group = 56

Dependent Variable: EXCH.Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.00229577	0.56	0.7611
Error	49	0.03357721		
Corrected Total	55	0.03587298		
R-Square		C.V.	EXCH.Mn Mean	
	0.063997	137.9047	0.01898214	

Source	DF	Type I SS	F Value	Pr > F
METH	6	0.00229577	0.56	0.7611
Source	DF	Type III SS	F Value	Pr > F
METH	6	0.00229577	0.56	0.7611

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 49 MSE= 0.000685
Critical Value of Studentized Range= 4.347

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means		Lower Confidence Limit	Upper Confidence Limit
9 - 1	-0.04113	0.00739	0.00739		-0.04113	0.05592
9 - 7	-0.05270	0.01300	0.01300		-0.05270	0.07870
9 - 5	-0.04837	0.01733	0.01733		-0.04837	0.08304
9 - 4	-0.03497	0.01800	0.01800		-0.03497	0.07097
9 - 3	-0.07292	0.02000	0.02000		-0.07292	0.11292
9 - 8	-0.03837	0.02733	0.02733		-0.03837	0.09304
1 - 9	-0.05592	-0.00739	-0.00739		-0.05592	0.04113
1 - 7	-0.04292	0.00561	0.00561		-0.04292	0.05413
1 - 5	-0.03859	0.00394	0.00394		-0.03859	0.05846
1 - 4	-0.01844	0.01061	0.01061		-0.01844	0.03965
1 - 3	-0.06907	0.01261	0.01261		-0.06907	0.09429
1 - 8	-0.02859	0.01994	0.01994		-0.02859	0.08846
7 - 9	-0.07870	-0.01300	-0.01300		-0.07870	0.05270
7 - 1	-0.05413	-0.00561	-0.00561		-0.05413	0.04292
7 - 5	-0.06137	0.00433	0.00433		-0.06137	0.07004
7 - 4	-0.04797	0.00500	0.00500		-0.04797	0.05797
7 - 3	-0.08592	0.00700	0.00700		-0.08592	0.09992
7 - 8	-0.05137	0.01433	0.01433		-0.05137	0.08004
5 - 9	-0.08304	-0.01733	-0.01733		-0.08304	0.04837
5 - 1	-0.05846	-0.00394	-0.00394		-0.05846	0.03859
5 - 7	-0.07004	-0.00433	-0.00433		-0.07004	0.06137
5 - 4	-0.05231	0.00067	0.00067		-0.05231	0.05364
5 - 3	-0.09025	0.00267	0.00267		-0.09025	0.09559
5 - 8	-0.05570	0.01000	0.01000		-0.05570	0.07570
4 - 9	-0.07097	-0.01800	-0.01800		-0.07097	0.03497
4 - 1	-0.03965	-0.01061	-0.01061		-0.03965	0.01844
4 - 7	-0.05797	-0.00500	-0.00500		-0.05797	0.04797
4 - 5	-0.05364	-0.00067	-0.00067		-0.05364	0.05231
4 - 3	-0.08240	0.00200	0.00200		-0.08240	0.08640
4 - 8	-0.04364	0.00933	0.00933		-0.04364	0.06231
3 - 9	-0.11292	-0.02000	-0.02000		-0.11292	0.07292
3 - 1	-0.09429	-0.01261	-0.01261		-0.09429	0.06907
3 - 7	-0.09992	-0.00700	-0.00700		-0.09992	0.08592
3 - 5	-0.09559	-0.00267	-0.00267		-0.09559	0.09025

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
3 - 4	-0.08640	-0.00200	0.08240	
3 - 8	-0.08559	0.00733	0.10025	
8 - 9	-0.09304	-0.02733	0.03837	
8 - 1	-0.06846	-0.01994	0.02859	
8 - 7	-0.08004	-0.01433	0.05137	
8 - 5	-0.07570	-0.01000	0.05570	
8 - 4	-0.06231	-0.00933	0.04364	
8 - 3	-0.10025	-0.00733	0.08559	

units: cmol+/kg
mandatory for samples B1 and B2
reported by 11 laboratories

TABLE I : Exchangeable H - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2			
3			
4	B1 B2	4	N/R
5	B1 B2	1 not reported	N/R
6			
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	B1 B2	3	R
10	B1 B2	3	N/R
11			
12			
13	A1 A2 B1 B2	3	N
14			
15			
16	B1 B2	3	N
17	B1 B2	3	N
18			
19	B1 B2	3	N=R
20			
21	B1 B2	3	R
22			

TABLE II : Exchangeable H - Summary method description

method	description
1	reference, 0.1M BaCl2 1:10
* 2	1N KCl
* 3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
* 6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
* 10	0.1M BaCl2
* 11	1M NH4Ac
12	unspecified

* not applicable

TABLE V : exch.H Method comparison

----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 11 4 5 7 8 9

Number of observations in by group = 41

Dependent Variable: EXCH.H

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	4.67098449	1.03	0.4206
Error	34	25.59930200		
Corrected Total	40	30.27028649		

R-Square	C.V.	EXCH.H Mean
0.154309	95.21749	0.91129268

Source	DF	Type I SS	F Value	Pr > F
METH	6	4.67098449	1.03	0.4206
Source	DF	Type III SS	F Value	Pr > F
METH	6	4.67098449	1.03	0.4206

TABLE IV : Exchangeable H - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A2	7	3	7.557	1.798	0.969	0.661
B1	1	21	1.077	1.080	0.721	0.000
B1	4	7	0.675	0.607	0.818	0.061
B1	5	3	0.213	0.021	0.923	0.463
B1	7	3	1.637	0.153	0.964	0.637
B1	8	3	0.667	0.068	0.912	0.424
B1	9	3	0.730	0.108	0.942	0.537
B1	11	1	0.290			
B2	1	21	1.424	1.374	0.675	0.000
B2	4	7	1.095	0.108	0.926	0.536
B2	5	3	0.750	0.056	0.976	0.702
B2	7	3	1.207	0.124	0.784	0.077
B2	8	3	0.540	0.035	0.750	0.000
B2	9	3	0.827	0.200	0.899	0.384
B2	11	1	0.600			

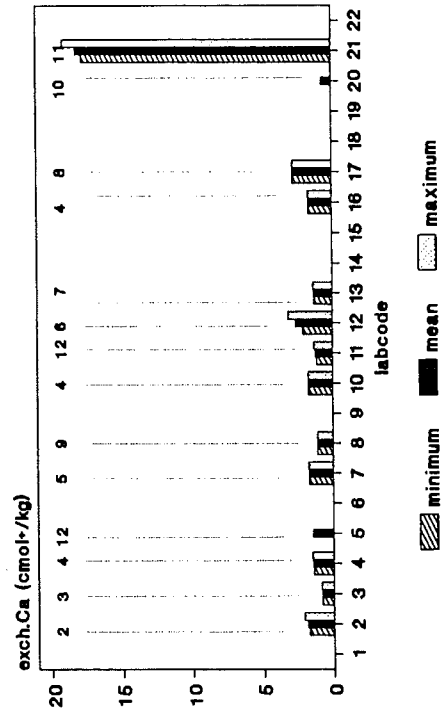
Remark:

Labcode 19 reported mean values for 3 replicates.

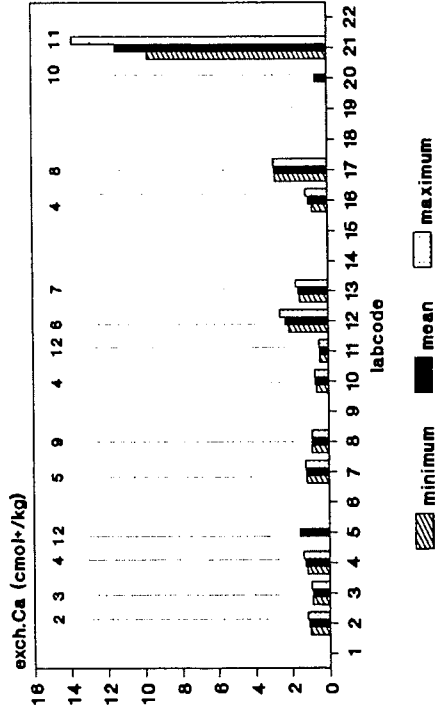
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.

So, the "repl" values in this table for method 1 are different from the real number of replicates.

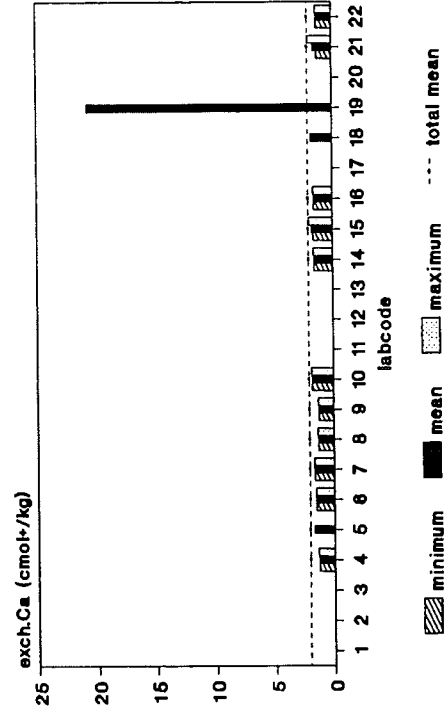
exch.Ca sample B1
national methods



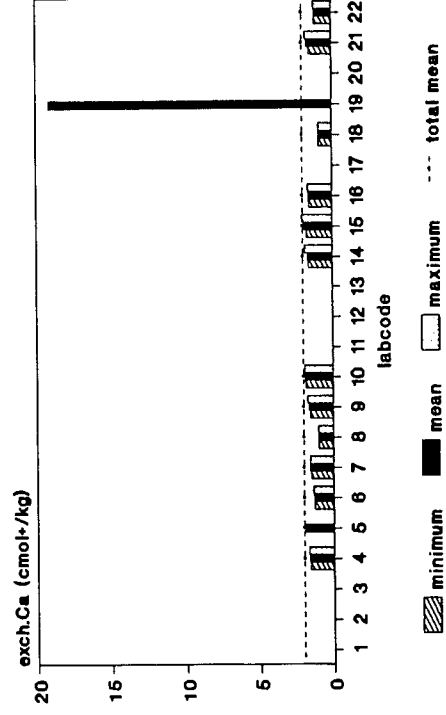
exch.Ca sample B2
national methods



exch.Ca sample B1
reference method



exch.Ca sample B2
reference method



SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.H

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 34 MSE= 1.116805
Critical Value of Studentized Range= 4.428

Number of observations in by group = 41

Comparisons significant at the 0.05 level are indicated by '****'.

Dependent Variable: EXCH.H				
Source	DF	Sum of Squares	F Value	Pr > F
Model	6	3.79579819	0.57	0.7539
Error	34	37.97136971		
Corrected Total	40	41.76716790		
R-Square C.V. EXCH.H Mean				
		90.01244		1.17404878
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	6	3.79579819	0.57	0.7539
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	6	3.79579819	0.57	0.7539

METH Comparison		Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit			
1	- 7	-1.825	2.260	0.217	2.260	
1	- 4	-1.115	1.774	0.329	1.774	
1	- 9	-1.445	2.640	0.597	2.640	
1	- 5	-1.369	2.716	0.674	2.716	
1	- 11	-2.563	4.211	0.824	4.211	
1	- 8	-1.159	2.926	0.884	2.926	
7	- 1	-2.260	1.825	-0.217	1.825	
7	- 4	-2.172	2.396	0.112	2.396	
7	- 9	-2.322	3.082	0.380	3.082	
7	- 5	-2.245	3.159	0.457	3.159	
7	- 11	-3.215	4.428	0.607	4.428	
7	- 8	-2.035	3.369	0.667	3.369	
4	- 1	-1.774	1.115	-0.329	1.115	
4	- 7	-2.396	2.172	-0.112	2.172	
4	- 9	-2.016	2.552	0.268	2.552	
4	- 5	-1.939	2.628	0.345	2.628	
4	- 11	-3.043	4.032	0.495	4.032	
4	- 8	-1.729	2.838	0.555	2.838	
9	- 1	-2.640	1.445	-0.597	1.445	
9	- 7	-3.082	2.322	-0.380	2.322	
9	- 4	-2.552	2.016	-0.268	2.016	
9	- 5	-2.625	2.779	0.077	2.779	
9	- 11	-3.595	4.048	0.227	4.048	
9	- 8	-2.415	2.989	0.287	2.989	
5	- 1	-2.716	1.369	-0.674	1.369	
5	- 7	-3.159	2.245	-0.457	2.245	
5	- 4	-2.628	1.939	-0.345	1.939	
5	- 9	-2.779	2.625	-0.077	2.625	
5	- 11	-3.671	3.971	0.150	3.971	
5	- 8	-2.492	2.912	0.210	2.912	
11	- 1	-4.211	2.563	-0.824	2.563	
11	- 7	-4.428	3.043	-0.607	3.043	
11	- 4	-4.032	3.015	-0.495	3.015	
11	- 9	-4.048	3.595	-0.227	3.595	

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
11 - 8	-3.514	-0.377	2.761	
11 - 5	-3.061	0.077	3.214	
5 - 7	-3.642	-1.423	0.795	
5 - 1	-2.540	-0.863	0.814	
5 - 9	-2.735	-0.517	1.702	
5 - 4	-2.336	-0.461	1.414	
5 - 8	-2.672	-0.453	1.765	
5 - 11	-3.214	-0.077	3.061	

Tukey's Studentized Range (HSD) Test for variable: EXCH.H

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 34 MSE= 0.752921

Critical Value of Studentized Range= 4.428

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
7 - 1	-1.117	0.560	2.237	
7 - 9	-1.312	0.907	3.125	
7 - 4	-0.913	0.962	2.837	
7 - 8	-1.249	0.970	3.189	
7 - 11	-1.791	1.347	4.484	
7 - 5	-0.795	1.423	3.642	
1 - 7	-2.237	-0.560	1.117	
1 - 9	-1.330	0.347	2.024	
1 - 4	-0.784	0.402	1.588	
1 - 8	-1.267	0.410	2.087	
1 - 11	-1.994	0.787	3.568	
1 - 5	-0.814	0.863	2.540	
9 - 7	-3.125	-0.907	1.312	
9 - 1	-2.024	-0.347	1.330	
9 - 4	-1.820	0.055	1.930	
9 - 8	-2.155	0.063	2.282	
9 - 11	-2.697	0.440	3.577	
9 - 5	-1.702	0.517	2.735	
4 - 7	-2.837	-0.962	0.913	
4 - 1	-1.588	-0.402	0.784	
4 - 9	-1.930	-0.055	1.820	
4 - 8	-1.867	0.008	1.883	
4 - 11	-2.520	0.385	3.289	
4 - 5	-1.414	0.461	2.336	
8 - 7	-3.189	-0.970	1.249	
8 - 1	-2.087	-0.410	1.267	
8 - 9	-2.282	-0.063	2.155	
8 - 4	-1.883	-0.008	1.867	
8 - 11	-2.761	0.377	3.514	
8 - 5	-1.765	0.453	2.672	
11 - 7	-4.484	-1.347	1.791	
11 - 1	-3.568	-0.787	1.994	
11 - 9	-3.577	-0.440	2.697	
11 - 4	-3.289	-0.385	2.520	

TABLE IIIa : Exchangeable H - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
13	A1	7	3	5.970	0.000	9.510	1.798	23.797
13	A2	7	3	5.970	7.557	9.510	1.798	23.797

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4	4	4	0.989	1.146	1.448	0.213	18.585
5	11	1		0.290			
6							
7	5	3	0.190	0.213	0.230	0.021	9.758
8	9	3	0.610	0.730	0.820	0.108	14.817
9							
10							
11							
12							
13	7	3	1.470	1.637	1.770	0.153	9.333
14							
15							
16	4	3	0.033	0.046	0.060	0.014	29.143
17	8	3	0.590	0.667	0.720	0.068	10.210
18							
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3							
4	4	4	1.112	1.174	1.248	0.059	5.058
5	11	1		0.600			
6							
7	5	3	0.700	0.750	0.810	0.056	7.424
8	9	3	0.600	0.827	0.980	0.200	24.234
9							
10							
11							
12							
13	7	3	1.130	1.207	1.350	0.124	10.295
14							
15							
16	4	3	0.966	0.989	1.010	0.022	2.228
17	8	3	0.520	0.540	0.580	0.035	6.415
18							
19							
20							
21							
22							

TABLE IIIb : Exchangeable H - descriptive statistics

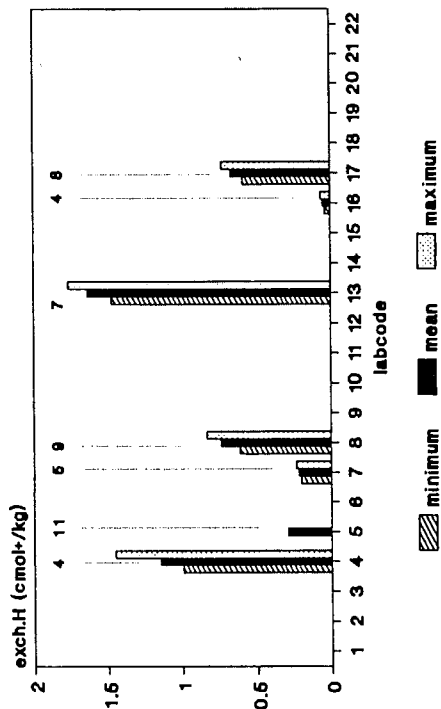
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.989	1.146	1.448	0.213	18.585
5	1		0.220			
6						
7	3	0.180	0.200	0.220	0.020	10.000
8	3	0.610	0.730	0.820	0.108	14.817
9	3	0.336	0.343	0.352	0.008	2.430
10	3	0.690	0.707	0.720	0.015	2.162
11						
12						
13						
14						
15						
16						
17						
18						
19	3		1.370			
20						
21	3	3.500	3.500	3.500	0.000	0.000
22						

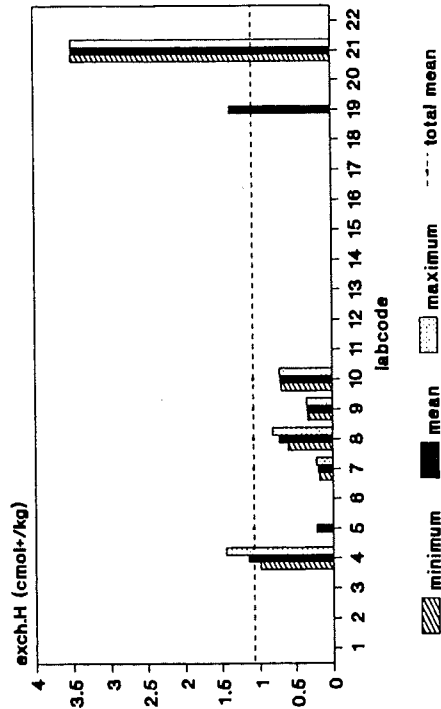
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	1.112	1.174	1.248	0.059	5.058
5	1		0.200			
6						
7	3	0.620	0.637	0.660	0.021	3.270
8	3	0.600	0.827	0.980	0.200	24.234
9	3	0.673	0.679	0.690	0.010	1.446
10	3	0.810	0.830	0.860	0.026	3.188
11						
12						
13						
14						
15						
16						
17						
18						
19	3		5.590			
20						
21	3	3.500	3.500	3.500	0.000	0.000
22						

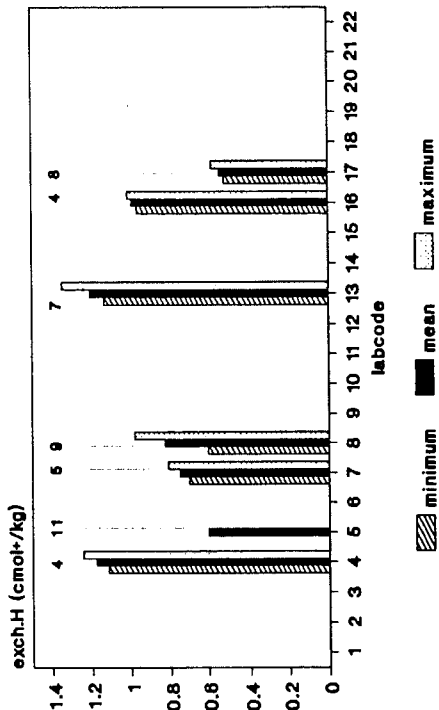
exch.H sample B1
national methods



exch.H sample B1
reference method



exch.H sample B2
national methods



exch.H sample B2
reference method

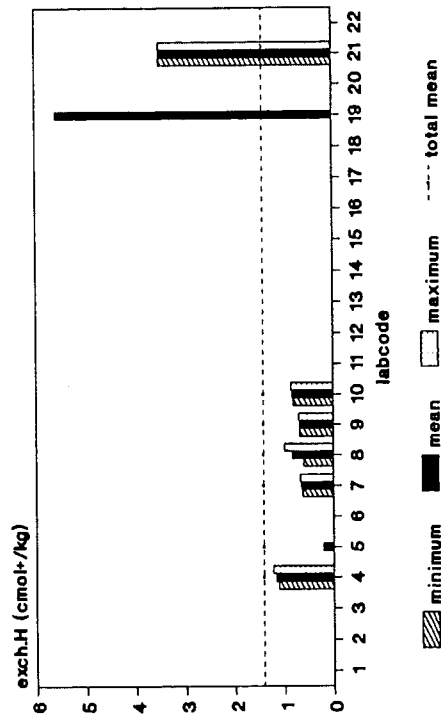


TABLE IIIa : Exchangeable Ca - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
2	A1	2	3	23.920	24.517	24.940	0.532	2.168
3	A1	3	5	16.270	16.421	16.540	0.609	
4	A1	4	4	21.270	21.450	21.580	0.140	0.654
13	A1	7	3	23.137	23.670	24.237	0.551	2.327
21	A1	11	3	71.300	75.400	80.900	4.951	6.566
3	A2	3	5	26.330	26.616	26.830	0.230	0.864
13	A2	7	3	28.140	30.951	35.161	3.714	11.998

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	1.680	1.830	2.080	0.218	11.910
2	3	5	0.810	0.824	0.840	0.010	1.214
3	4	4	1.373	1.415	1.476	0.047	3.302
4	12	1		1.450			
6	5	3	1.650	1.670	1.690	0.020	1.198
7	9	3	1.090	1.090	1.090	0.000	0.000
9	4	3	1.690	1.703	1.710	0.012	0.678
10	12	3	1.140	1.223	1.290	0.076	6.243
11	6	7	2.060	2.577	3.160	0.367	14.246
12	7	3	1.240	1.303	1.349	0.056	4.322
13							
14							
15	4	3	1.600	1.630	1.660	0.030	1.840
16	8	3	2.760	2.770	2.780	0.010	0.361
17							
18							
19							
20	10	1		0.661			
21	11	3	17.800	18.233	19.100	0.751	4.116
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	1.020	1.103	1.170	0.076	6.922
2	3	5	0.880	0.902	0.940	0.020	2.217
3	4	4	1.146	1.270	1.361	0.096	7.582
4	12	1		1.580			
6	5	3	1.150	1.193	1.220	0.038	3.173
7	9	3	0.890	0.890	0.890	0.000	0.000
9	4	3	0.620	0.670	0.700	0.044	6.506
10	12	3	0.440	0.457	0.480	0.021	4.558
11	6	6	2.120	2.338	2.640	0.188	8.035
12	7	3	1.517	1.605	1.750	0.126	7.868
13							
14							
15	4	3	0.830	1.023	1.170	0.175	17.075
16	8	3	2.830	2.877	2.910	0.042	1.447
17							
18							
19							
20	10	1		0.629			
21	11	3	9.750	11.450	13.800	2.102	18.356
22							

TABLE IIIb : Exchangeable Ca - descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
4	4	19.620	19.920	20.170	0.272	1.366
9	3	17.800	18.067	18.300	0.252	1.393
14	5	18.160	18.376	18.560	0.148	0.804
19	3		201.000			
21	3	25.600	26.200	26.600	0.529	2.020

sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	1.316	1.341	1.367	0.024	1.756
5	1		1.700			
6	2	1.530	1.530	1.530	0.000	0.000
7	3	1.670	1.697	1.720	0.025	1.483
8	3	1.320	1.353	1.370	0.029	2.133
9	3	1.270	1.300	1.320	0.026	2.035
10	3	1.820	1.827	1.840	0.012	0.632
11						
12						
13						
14	5	1.574	1.612	1.633	0.026	1.637
15	3	1.650	1.797	2.030	0.204	11.370
16	3	1.580	1.603	1.630	0.025	1.570
17						
18	1		1.800			
19	3		20.800			
20						
21	3	1.280	1.620	2.050	0.393	24.248
22	3	1.336	1.353	1.366	0.015	1.129

sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	1.564	1.643	1.714	0.080	4.844
5	1		1.940			
6	2	1.300	1.335	1.370	0.049	3.708
7	3	1.510	1.563	1.600	0.047	3.023
8	3	1.000	1.007	1.020	0.012	1.147
9	3	1.530	1.653	1.730	0.108	6.524
10	3	1.820	1.867	1.930	0.057	3.046
11						
12						
13						
14	5	1.626	1.687	1.895	0.117	6.911
15	3	1.740	1.893	2.030	0.146	7.696
16	3	1.580	1.610	1.650	0.036	2.239
17						
18	1	0.910	0.910	0.910		
19	3		19.100			
20						
21	3	1.530	1.697	1.780	0.144	8.507
22	3	1.146	1.169	1.198	0.026	2.258

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
11 - 5	-3.971	-0.150	3.671	
11 - 8	-3.761	0.060	3.881	
8 - 1	-2.926	-0.884	1.159	
8 - 7	-3.369	-0.667	2.035	
8 - 4	-2.838	-0.555	1.729	
8 - 9	-2.989	-0.287	2.415	
8 - 5	-2.912	-0.210	2.492	
8 - 11	-3.881	-0.060	3.761	

14. EXCHANGEABLE Ca

units: cmol+/kg
mandatory for samples B1 and B2
reported by 21 laboratories

TABLE I : Exchangeable Ca - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2	A1 B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	A1 B1 B2	4	N/R
5	B1 B2	1 not reported	N/R
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	A1 B1 B2	3	R
10	B1 B2	3	N/R
11	B1 B2	3	N
12	B1 B2	B1:7 B2:6	N
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N=R
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	1 not reported	N=R
19	A1 B1 B2	3	N=R
20	B1 B2	1 not reported	N
21	A1 B1 B2	3	N/R
22	B1 B2	3	R

TABLE II : Exchangeable Ca - Summary method descriptions

method	description
1	reference, 0.1M BaCl2 1:10
2	1N NH4Ac at pH 7
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
11	1M NH4Ac
12	unspecified

TABLE V : exch.Ca Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 11 2 3 4 7

Number of observations in by group = 30

Dependent Variable: EXCH.Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	6706.30529830	1.04	0.4145
Error	24	30804.59740067		
Corrected Total	29	37510.90269897		

R-Square	C.V.	EXCH.Ca Mean
0.178783	109.8584	32.61136667

Source	DF	Type I SS	F Value	Pr > F
METH	5	6706.30529830	1.04	0.4145

Source	DF	Type III SS	F Value	Pr > F
METH	5	6706.30529830	1.04	0.4145

TABLE IV : Exchangeable Ca - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	16	31.585	45.280	0.320	0.000
A1	2	3	24.517	0.532	0.920	0.453
A1	3	1	16.421			
A1	4	4	21.450	0.140	0.935	0.601
A1	7	3	23.670	0.551	0.997	0.898
A1	11	3	75.400	4.951	0.940	0.527
A2	3	1	26.616			
A2	7	3	30.951	3.714	0.894	0.366
B1	1	38	2.064	3.128	0.215	0.000
B1	2	3	1.830	0.218	0.842	0.220
B1	3	1	0.824			
B1	4	10	1.566	0.137	0.866	0.086
B1	5	3	1.670	0.020	1.000	0.992
B1	6	7	2.577	0.367	0.941	0.666
B1	7	3	1.303	0.056	0.937	0.515
B1	8	3	2.770	0.010	1.000	0.992
B1	9	3	1.090	0.000	0.000	0.000
B1	10	1	0.661			
B1	11	3	18.233	0.751	0.750	0.000
B1	12	4	1.280	0.129	0.977	0.852
B2	1	38	2.026	2.859	0.244	0.000
B2	2	3	1.103	0.076	0.964	0.637
B2	3	1	0.902			
B2	4	10	1.016	0.281	0.899	0.204
B2	5	3	1.193	0.038	0.855	0.253
B2	6	6	2.338	0.188	0.949	0.752
B2	7	3	1.605	0.126	0.851	0.243
B2	8	3	2.877	0.042	0.923	0.463
B2	9	3	0.890	0.000	0.000	0.000
B2	10	1	0.629			
B2	11	3	11.450	2.102	0.928	0.482
B2	12	4	0.737	0.562	0.656	0.002

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates. To calculate the total number of replicates for the method groups, SAS counts the number of reported results. So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

exch.Ca Method comparison

exch.Ca Method comparison

SAMPLE=A1

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure
Class Level Information

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 24 MSE= 1283.525

Critical Value of Studentized Range= 4.373

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	
11 - 1	-25.88	113.51	43.81	113.51	
11 - 2	-39.56	141.33	50.88	141.33	
11 - 7	-38.72	142.18	51.73	142.18	
11 - 4	-30.65	138.55	53.95	138.55	
11 - 3	-68.93	186.89	58.98	186.89	
1 - 11	-113.51	25.88	-43.81	25.88	
1 - 2	-62.62	76.76	7.07	76.76	
1 - 7	-61.78	77.61	7.92	77.61	
1 - 4	-51.79	72.06	10.14	72.06	
1 - 3	-99.02	129.35	15.16	129.35	
2 - 11	-141.33	39.56	-50.88	39.56	
2 - 1	-76.76	62.62	-7.07	62.62	
2 - 7	-89.60	91.29	0.85	91.29	
2 - 4	-81.54	87.67	3.07	87.67	
2 - 3	-119.81	136.01	8.10	136.01	
7 - 11	-142.18	38.72	-51.73	38.72	
7 - 1	-77.61	61.78	-7.92	61.78	
7 - 2	-91.29	89.60	-0.85	89.60	
7 - 4	-82.38	86.82	2.22	86.82	
7 - 3	-120.66	135.16	7.25	135.16	
4 - 11	-138.55	30.65	-53.95	30.65	
4 - 1	-72.06	51.79	-10.14	51.79	
4 - 2	-87.67	81.54	-3.07	81.54	
4 - 7	-86.82	82.38	-2.22	82.38	
4 - 3	-118.82	128.88	5.03	128.88	
3 - 11	-186.89	68.93	-58.98	68.93	
3 - 1	-129.35	99.02	-15.16	99.02	
3 - 2	-136.01	119.81	-8.10	119.81	
3 - 7	-135.16	120.66	-7.25	120.66	
3 - 4	-128.88	118.82	-5.03	118.82	

Dependent Variable: EXCH.Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	14.09633633	1.02	0.4184
Error	2	27.58022467		
Corrected Total	3	41.67656100		
R-Square				
		C.V.	EXCH.Ca Mean	
		0.338232	12.43326	29.86750000

Source	DF	Type I SS	F Value	Pr > F
METH	1	14.09633633	1.02	0.4184
Source	DF	Type III SS	F Value	Pr > F
METH	1	14.09633633	1.02	0.4184

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Class Levels Values

METH 12 1 10 11 12 2 3 4 5 6 7 8 9

Number of observations in by group = 79

Dependent Variable: EXCH.Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	784.59041481	13.12	0.0001
Error	67	364.37818055		
Corrected Total	78	1148.96859537		

R-Square C.V. EXCH.Ca Mean

0.682865 92.37945 2.52443038

Source	DF	Type I SS	F Value	Pr > F
METH	11	784.59041481	13.12	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	11	784.59041481	13.12	0.0001

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 67 MSE= 5.43848

Critical Value of Studentized Range= 4.789

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
11 - 8	9.016	21.911	15.463	9.016	21.911
11 - 6	10.207	21.105	15.656	10.207	21.105
11 - 1	11.434	20.905	16.169	11.434	20.905
11 - 2	9.956	22.851	16.403	9.956	22.851
11 - 5	10.116	23.011	16.563	10.116	23.011
11 - 4	11.469	21.865	16.667	11.469	21.865
11 - 7	10.483	23.378	16.931	10.483	23.378
11 - 12	10.922	22.984	16.953	10.922	22.984
11 - 9	10.696	23.591	17.143	10.696	23.591
11 - 3	8.291	26.528	17.409	8.291	26.528
11 - 10	8.454	26.691	17.572	8.454	26.691
8 - 11	-21.911	-9.016	-15.463	-21.911	-9.016
8 - 6	-5.256	5.642	0.193	-5.256	5.642
8 - 1	-4.030	5.442	0.706	-4.030	5.442
8 - 2	-5.508	7.388	0.940	-5.508	7.388
8 - 5	-5.348	1.100	1.100	-5.348	1.100
8 - 4	-3.994	1.204	1.204	-3.994	1.204
8 - 7	-4.980	1.467	1.467	-4.980	1.467
8 - 12	-4.541	1.490	1.490	-4.541	1.490
8 - 9	-4.768	1.680	1.680	-4.768	1.680
8 - 3	-7.172	1.946	1.946	-7.172	1.946
8 - 10	-7.009	2.109	2.109	-7.009	2.109
6 - 11	-21.105	-10.207	-15.656	-21.105	-10.207
6 - 8	-5.642	5.256	-0.193	-5.642	5.256
6 - 1	-2.735	3.761	0.513	-2.735	3.761
6 - 2	-4.702	6.196	0.747	-4.702	6.196
6 - 5	-4.542	6.356	0.907	-4.542	6.356
6 - 4	-2.881	1.011	1.011	-2.881	1.011
6 - 7	-4.175	1.274	1.274	-4.175	1.274
6 - 12	-3.652	1.297	1.297	-3.652	1.297
6 - 9	-3.962	1.487	1.487	-3.962	1.487
6 - 3	-6.689	1.753	1.753	-6.689	1.753
6 - 10	-6.526	1.916	1.916	-6.526	1.916
1 - 11	-20.905	-11.434	-16.169	-20.905	-11.434
1 - 8	-5.442	4.030	-0.706	-5.442	4.030
1 - 6	-3.761	2.735	-0.513	-3.761	2.735

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 1	-5.497	3.974	-0.761	-16.953	-10.922
7 - 2	-6.975	5.920	-0.527	-1.490	4.541
7 - 5	-6.815	6.080	-0.367	-1.297	3.652
7 - 4	-5.462	4.935	-0.264	-0.784	3.367
7 - 12	-6.008	6.054	0.023	-0.550	5.481
7 - 9	-6.235	6.660	0.213	-0.390	5.641
7 - 3	-8.640	9.597	0.479	-0.286	4.386
7 - 10	-8.477	9.760	0.642	-0.023	6.008
				0.190	6.221
				0.456	9.285
				0.619	9.448

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 2	-4.502	4.970	0.234	-16.403	-9.956
1 - 5	-4.342	5.130	0.394	-0.940	5.508
1 - 4	-2.309	3.304	0.498	-0.747	4.702
1 - 7	-3.974	5.497	0.761	-0.234	4.502
1 - 12	-3.367	4.935	0.784	0.160	6.608
1 - 9	-3.762	5.710	0.974	0.264	5.462
1 - 3	-6.760	9.240	1.240	0.527	6.975
1 - 10	-6.597	9.403	1.403	0.550	6.581
				0.740	7.188
				1.006	10.124
				1.169	10.287

11

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7

12

----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
10 - 1	-9.403	-1.403	6.597	
10 - 2	-10.287	-1.169	7.949	
10 - 5	-10.127	-1.009	8.109	
10 - 4	-9.187	-0.905	7.377	
10 - 7	-9.760	-0.642	8.477	
10 - 12	-9.448	-0.619	8.210	
10 - 9	-9.547	-0.429	8.689	
10 - 3	-11.331	-0.163	11.005	

Number of observations in by group = 78

Dependent Variable: EXCH.Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	297.96400464	5.71	0.0001
Error	66	313.16039008		
Corrected Total	77	611.12439472		
R-Square				
C.V.				
EXCH.Ca Mean				
0.487567				
104.2170				
2.09012821				

Source	DF	Type I SS	F Value	Pr > F
METH	11	297.96400464	5.71	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	11	297.96400464	5.71	0.0001

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca
 NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 4.744854
 Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 8	2.548	8.573	14.599	14.599	***	***
11 - 6	3.893	9.112	14.330	14.330	***	***
11 - 1	4.998	9.424	13.849	13.849	***	***
11 - 7	3.819	9.845	15.870	15.870	***	***
11 - 5	4.231	10.257	16.282	16.282	***	***
11 - 2	4.321	10.347	16.372	16.372	***	***
11 - 4	5.576	10.434	15.292	15.292	***	***
11 - 3	2.026	10.548	19.070	19.070	***	***
11 - 9	4.534	10.560	16.586	16.586	***	***
11 - 12	5.076	10.713	16.349	16.349	***	***
11 - 10	2.299	10.821	19.343	19.343	***	***
8 - 11	-14.599	-8.573	-2.548	-2.548	***	***
8 - 6	-4.680	0.538	5.757	5.757		
8 - 1	-3.576	0.850	5.276	5.276		
8 - 7	-4.754	1.271	7.297	7.297		
8 - 5	-4.342	1.683	7.709	7.709		
8 - 2	-4.252	1.773	7.799	7.799		
8 - 4	-2.997	1.861	6.719	6.719		
8 - 3	-6.547	1.975	10.496	10.496		
8 - 9	-4.039	1.987	8.012	8.012		
8 - 12	-3.497	2.139	7.776	7.776		
8 - 10	-6.274	2.248	10.769	10.769		
6 - 11	-14.330	-9.112	-3.893	-3.893	***	***
6 - 8	-5.757	-0.538	4.680	4.680		
6 - 1	-2.930	0.312	3.554	3.554		
6 - 7	-4.485	0.733	5.951	5.951		
6 - 5	-4.073	1.145	6.363	6.363		
6 - 2	-3.983	1.235	6.453	6.453		
6 - 4	-2.489	1.322	5.133	5.133		
6 - 3	-6.535	1.436	9.408	9.408		
6 - 9	-3.770	1.448	6.667	6.667		
6 - 12	-3.163	1.601	6.365	6.365		
6 - 10	-6.262	1.709	9.681	9.681		
1 - 11	-13.849	-9.424	-4.998	-4.998	***	***
1 - 8	-5.276	-0.850	3.576	3.576		
1 - 6	-3.554	-0.312	2.930	2.930		

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca
 NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 4.744854
 Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 8	2.548	8.573	14.599	14.599	***	***
11 - 6	3.893	9.112	14.330	14.330	***	***
11 - 1	4.998	9.424	13.849	13.849	***	***
11 - 7	3.819	9.845	15.870	15.870	***	***
11 - 5	4.231	10.257	16.282	16.282	***	***
11 - 2	4.321	10.347	16.372	16.372	***	***
11 - 4	5.576	10.434	15.292	15.292	***	***
11 - 3	2.026	10.548	19.070	19.070	***	***
11 - 9	4.534	10.560	16.586	16.586	***	***
11 - 12	5.076	10.713	16.349	16.349	***	***
11 - 10	2.299	10.821	19.343	19.343	***	***
8 - 11	-14.599	-8.573	-2.548	-2.548	***	***
8 - 6	-4.680	0.538	5.757	5.757		
8 - 1	-3.576	0.850	5.276	5.276		
8 - 7	-4.754	1.271	7.297	7.297		
8 - 5	-4.342	1.683	7.709	7.709		
8 - 2	-4.252	1.773	7.799	7.799		
8 - 4	-2.997	1.861	6.719	6.719		
8 - 3	-6.547	1.975	10.496	10.496		
8 - 9	-4.039	1.987	8.012	8.012		
8 - 12	-3.497	2.139	7.776	7.776		
8 - 10	-6.274	2.248	10.769	10.769		
6 - 11	-14.330	-9.112	-3.893	-3.893	***	***
6 - 8	-5.757	-0.538	4.680	4.680		
6 - 1	-2.930	0.312	3.554	3.554		
6 - 7	-4.485	0.733	5.951	5.951		
6 - 5	-4.073	1.145	6.363	6.363		
6 - 2	-3.983	1.235	6.453	6.453		
6 - 4	-2.489	1.322	5.133	5.133		
6 - 3	-6.535	1.436	9.408	9.408		
6 - 9	-3.770	1.448	6.667	6.667		
6 - 12	-3.163	1.601	6.365	6.365		
6 - 10	-6.262	1.709	9.681	9.681		
1 - 11	-13.849	-9.424	-4.998	-4.998	***	***
1 - 8	-5.276	-0.850	3.576	3.576		
1 - 6	-3.554	-0.312	2.930	2.930		

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca
 NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 4.744854
 Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 8	2.548	8.573	14.599	14.599	***	***
11 - 6	3.893	9.112	14.330	14.330	***	***
11 - 1	4.998	9.424	13.849	13.849	***	***
11 - 7	3.819	9.845	15.870	15.870	***	***
11 - 5	4.231	10.257	16.282	16.282	***	***
11 - 2	4.321	10.347	16.372	16.372	***	***
11 - 4	5.576	10.434	15.292	15.292	***	***
11 - 3	2.026	10.548	19.070	19.070	***	***
11 - 9	4.534	10.560	16.586	16.586	***	***
11 - 12	5.076	10.713	16.349	16.349	***	***
11 - 10	2.299	10.821	19.343	19.343	***	***
8 - 11	-14.599	-8.573	-2.548	-2.548	***	***
8 - 6	-4.680	0.538	5.757	5.757		
8 - 1	-3.576	0.850	5.276	5.276		
8 - 7	-4.754	1.271	7.297	7.297		
8 - 5	-4.342	1.683	7.709	7.709		
8 - 2	-4.252	1.773	7.799	7.799		
8 - 4	-2.997	1.861	6.719	6.719		
8 - 3	-6.547	1.975	10.496	10.496		
8 - 9	-4.039	1.987	8.012	8.012		
8 - 12	-3.497	2.139	7.776	7.776		
8 - 10	-6.274	2.248	10.769	10.769		
6 - 11	-14.330	-9.112	-3.893	-3.893	***	***
6 - 8	-5.757	-0.538	4.680	4.680		
6 - 1	-2.930	0.312	3.554	3.554		
6 - 7	-4.485	0.733	5.951	5.951		
6 - 5	-4.073	1.145	6.363	6.363		
6 - 2	-3.983	1.235	6.453	6.453		
6 - 4	-2.489	1.322	5.133	5.133		
6 - 3	-6.535	1.436	9.408	9.408		
6 - 9	-3.770	1.448	6.667	6.667		
6 - 12	-3.163	1.601	6.365	6.365		
6 - 10	-6.262	1.709	9.681	9.681		
1 - 11	-13.849	-9.424	-4.998	-4.998	***	***
1 - 8	-5.276	-0.850	3.576	3.576		
1 - 6	-3.554	-0.312	2.930	2.930		

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca
 NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 4.744854
 Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 8	2.548	8.573	14.599	14.599	***	***
11 - 6	3.893	9.112	14.330	14.330	***	***
11 - 1	4.998	9.424	13.849	13.849	***	***
11 - 7	3.819	9.845	15.870	15.870	***	***
11 - 5	4.231	10.257	16.282	16.282	***	***
11 - 2	4.321	10.347	16.372	16.372	***	***
11 - 4	5.576	10.434	15.292	15.292	***	***
11 - 3	2.026	10.548	19.070	19.070	***	***
11 - 9	4.534	10.560	16.586	16.586	***	***
11 - 12	5.076	10.713	16.349	16.349	***	***
11 - 10	2.299	10.821	19.343	19.343	***	***
8 - 11	-14.599	-8.573	-2.548	-2.548	***	***
8 - 6	-4.680	0.538	5.757	5.757		
8 - 1	-3.576	0.850	5.276	5.276		
8 - 7	-4.754	1.271	7.297	7.297		
8 - 5	-4.342	1.683	7.709	7.709		
8 - 2	-4.252	1.773	7.799	7.799		
8 - 4	-2.997	1.861	6.719	6.719		
8 - 3	-6.547	1.975	10.496	10.496		
8 - 9	-4.039	1.987	8.012	8.012		
8 - 12	-3.497	2.139	7.776	7.776		
8 - 10	-6.274	2.248	10.769	10.769		
6 - 11	-14.330	-9.112	-3.893	-3.893	***	***
6 - 8	-5.757	-0.538	4.680	4.680		
6 - 1	-2.930	0.312	3.554	3.554		
6 - 7	-4.485	0.733	5.951	5.951		
6 - 5	-4.073	1.145	6.363	6.363		
6 - 2	-3.983	1.235	6.453	6.453		
6 - 4	-2.489	1.322	5.133	5.133		
6 - 3	-6.535	1.436	9.408	9.408		
6 - 9	-3.770	1.448	6.667	6.667		
6 - 12	-3.163	1.601	6.365	6.365		
6 - 10	-6.262	1.709	9.681	9.681		
1 - 11	-13.849	-9.424	-4.998	-4.998	***	***
1 - 8	-5.276	-0.850	3.576	3.576		
1 - 6	-3.554	-0.312	2.930	2.930		

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Ca
 NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 4.744854
 Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 8	2.548	8.573	14.599	*	*
1 - 6	3.893	9.112	14.330	*	*
1 - 1	4.998	9.424	13.849	*	*
1 - 7	3.819	9.845	15.870	*	*
1 - 5	4.231	10.257	16.282	*	*
1 - 2	4.321	10.347	16.372	*	*
1 - 4	5.576	10.434	15.292	*	*
1 - 3	2.026	10.548	19.070	*	*
1 - 9	4.534	10.560	16.586	*	*
1 - 12	5.076	10.713	16.349	*	*
1 - 10	2.299	10.821	19.343	*	*
3 - 11	-14.599	-8.573	-2.548	*	*
3 - 6	-4.680	0.538	5.757		
3 - 1	-3.576	0.850	5.276		
3 - 7	-4.754	1.271	7.297		
3 - 5	-4.342	1.683	7.709		
3 - 2	-4.252	1.773	7.799		
3 - 4	-2.997	1.861	6.719		
3 - 3	-6.547	1.975	10.496		
3 - 9	-4.039	1.987	8.012		
3 - 12	-3.497	2.139	7.776		
3 - 10	-6.274	2.248	10.769		
5 - 11	-14.330	-9.112	-3.893	*	*
5 - 8	-5.757	-0.538	4.680		
5 - 1	-2.930	0.312	3.554		
5 - 7	-4.485	0.733	5.951		
5 - 5	-4.073	1.145	6.363		
5 - 2	-3.983	1.235	6.453		
5 - 4	-2.489	1.322	5.133		
5 - 3	-6.535	1.436	9.408		
5 - 9	-3.770	1.448	6.667		
5 - 12	-3.163	1.601	6.365		
5 - 10	-6.262	1.709	9.681		
11 - 1	-13.849	-9.424	-4.998	*	*
11 - 8	-5.276	-0.850	3.576		
11 - 6	-3.554	-0.312	2.930		

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
10 - 1	-8.874	-1.397	-1.397	-8.874	6.079
10 - 7	-9.498	-0.976	-0.976	-9.498	7.545
10 - 5	-9.086	-0.564	-0.564	-9.086	7.957
10 - 2	-8.996	-0.474	-0.474	-8.996	8.047
10 - 4	-8.127	-0.387	-0.387	-8.127	7.353
10 - 3	-10.710	-0.273	-0.273	-10.710	10.164
10 - 9	-8.783	-0.261	-0.261	-8.783	8.261
10 - 12	-8.360	-0.109	-0.109	-8.360	8.143

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous		
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit	
4 - 1	-3.633	1.613	-1.010	-3.633	1.613	
4 - 7	-5.447	4.269	-0.589	-5.447	4.269	
4 - 5	-5.035	4.681	-0.177	-5.035	4.681	
4 - 2	-4.945	4.771	-0.087	-4.945	4.771	
4 - 3	-7.626	7.854	0.114	-7.626	7.854	
4 - 9	-4.732	4.984	0.126	-4.732	4.984	
4 - 12	-4.088	4.645	0.278	-4.088	4.645	
4 - 10	-7.353	8.127	0.387	-7.353	8.127	
3 - 11	-19.070	-2.026	-10.548	-19.070	-2.026	***
3 - 8	-10.496	6.547	-1.975	-10.496	6.547	
3 - 6	-9.408	6.535	-1.436	-9.408	6.535	
3 - 1	-8.601	6.352	-1.124	-8.601	6.352	
3 - 7	-9.225	7.818	-0.703	-9.225	7.818	
3 - 5	-8.813	8.230	-0.291	-8.813	8.230	
3 - 2	-8.723	8.320	-0.201	-8.723	8.320	
3 - 4	-7.854	7.626	-0.114	-7.854	7.626	
3 - 9	-8.510	8.534	0.012	-8.510	8.534	
3 - 12	-8.087	8.416	0.164	-8.087	8.416	
3 - 10	-10.164	10.710	0.273	-10.164	10.710	
9 - 11	-16.586	-4.534	-10.560	-16.586	-4.534	***
9 - 8	-8.012	4.039	-1.987	-8.012	4.039	
9 - 6	-6.667	3.770	-1.448	-6.667	3.770	
9 - 1	-5.562	3.289	-1.136	-5.562	3.289	
9 - 7	-6.741	5.310	-0.715	-6.741	5.310	
9 - 5	-6.329	5.722	-0.303	-6.329	5.722	
9 - 2	-6.239	5.812	-0.213	-6.239	5.812	
9 - 4	-4.984	4.732	-0.126	-4.984	4.732	
9 - 3	-8.534	8.510	-0.012	-8.534	8.510	
9 - 12	-5.484	5.789	0.152	-5.484	5.789	
9 - 10	-8.261	8.783	0.261	-8.261	8.783	
12 - 11	-16.349	-5.076	-10.713	-16.349	-5.076	***
12 - 8	-7.776	3.497	-2.139	-7.776	3.497	
12 - 6	-6.365	3.163	-1.601	-6.365	3.163	
12 - 1	-5.168	2.590	-1.289	-5.168	2.590	
12 - 7	-6.504	4.769	-0.868	-6.504	4.769	
12 - 5	-6.092	5.181	-0.456	-6.092	5.181	
12 - 2	-6.002	5.271	-0.366	-6.002	5.271	
12 - 4	-4.645	4.088	-0.278	-4.645	4.088	
12 - 3	-8.416	8.087	-0.164	-8.416	8.087	
12 - 9	-5.789	5.484	-0.152	-5.789	5.484	
12 - 10	-8.143	8.360	0.109	-8.143	8.360	
10 - 11	-19.343	-2.299	-10.821	-19.343	-2.299	***
10 - 8	-10.769	6.274	-2.248	-10.769	6.274	
10 - 6	-9.681	6.262	-1.709	-9.681	6.262	

15. EXCHANGEABLE Mg

units: cmol+/kg
mandatory for samples B1 and B2
reported by 21 laboratories

TABLE I : Exchangeable Mg - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1	A1 B1 B2	3	N
2	A1 A2 B1 B2	5	N
3	A1 B1 B2	4	N/R
4	A1 B1 B2	1 not reported	N/R
5	B1 B2	2	R
6	B1 B2	3	N/R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	A1 B1 B2	3	R
10	B1 B2	3	N/R
11	B1 B2	3	N
12	B1 B2	B1:7 B2:6	N
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N=R
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	1 not reported	N=R
19	A1 B1 B2	3	N=R
20	B1 B2	1 not reported	N
21	A1 B1 B2	3	N/R
22	B1 B2	3	R

TABLE II : Exchangeable Mg - Summary method description

method	description
1	reference, 0.1M BaCl2 1:10
2	1N NH4Ac at pH 7
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
11	1M NH4Ac
12	unspecified

TABLE IIIa : Exchangeable Mg - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
2	A1	2	3	2.21	2.237	2.29	0.046	2.065
3	A1	3	5	1.094	1.098	1.105	0.004	0.364298
4	A1	4	4	0.972	0.98	0.988	0.007	0.687
13	A1	7	3	1.84	1.863	1.883	0.022	1.162
21	A1	11	3	5.03	5.45	5.87	0.42	7.706
3	A2	3	5	3.683	3.724	3.748	0.028	0.751879
13	A2	7	3	5.032	5.186	5.359	0.164	3.167

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	1.02	1.047	1.1	0.046	4.413
2	3	5	0.483	0.486	0.486	0.001	0.206185
3	4	4	0.415	0.417	0.42	0.002	0.518
5	12	1		0.85			
6							
7	5	3	0.958	0.966	0.975	0.009	0.88
8	9	3	0.36	0.413	0.45	0.047	11.433
9							
10	4	3	1.02	1.027	1.03	0.006	0.562
11	12	3	0.92	0.943	0.96	0.021	2.207
12	6	7	0.47	0.604	0.72	0.084	13.972
13	7	3	0.803	0.812	0.82	0.009	1.048
14							
15							
16	4	3	0.9	0.947	0.97	0.04	4.269
17	8	3	1.7	1.7	1.7	0	0
18							
19							
20	10	1		0.383			
21	11	3	3.78	4.333	4.61	0.479	11.058
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	0.68	0.68	0.68	0	0
3	3	5	0.408	0.418	0.428	0.008	1.913875
4	4	4	0.306	0.328	0.348	0.02	6.158
5	12	1		0.7			
6							
7	5	3	0.594	0.617	0.632	0.02	3.249
8	9	3	0.35	0.357	0.36	0.006	1.619
9							
10	4	3	0.31	0.333	0.35	0.021	6.245
11	12	3	0.18	0.197	0.21	0.015	7.767
12	6	6	0.33	0.473	0.7	0.127	26.912
13	7	3	0.733	0.792	0.9	0.094	11.864
14							
15							
16	4	3	0.4	0.447	0.47	0.04	9.048
17	8	3	1.47	1.487	1.5	0.015	1.027
18							
19							
20	10	1		0.296			
21	11	3	2.1	2.65	3.34	0.632	23.839
22							

TABLE IIIb : Exchangeable Mg - descriptive statistics

sample A1 - reference method

labcode	sample	repl	minimum	mean	maximum	std	cv
4	A1	4	1.026	1.033	1.045	0.009	0.860
14	A1	5	2.130	2.149	2.166	0.017	0.787
19	A1	1	21.200	21.200	21.200		
21	A1	3	2.000	2.037	2.080	0.040	1.984

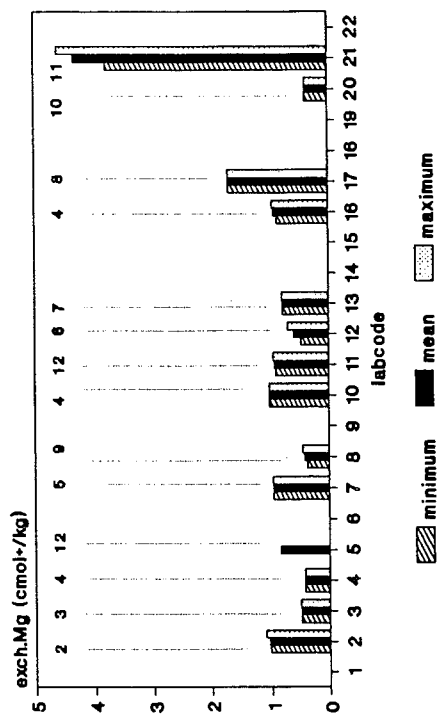
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.396	0.400	0.403	0.003	0.719
5	1		1.050			
6	2	0.830	0.875	0.920	0.064	7.273
7	3	0.962	0.975	0.989	0.014	1.393
8	3	0.400	0.407	0.420	0.012	2.839
9	3	0.820	0.837	0.860	0.021	2.488
10	3	1.060	1.063	1.070	0.006	0.543
11						
12						
13						
14	5	0.954	0.966	0.978	0.010	1.042
15	3	0.800	0.873	0.940	0.070	8.042
16	3	0.950	0.957	0.960	0.006	0.604
17						
18	1		0.995			
19	3		11.100			
20						
21	3	0.810	0.863	0.890	0.046	5.350
22	3	0.830	0.836	0.840	0.005	0.633

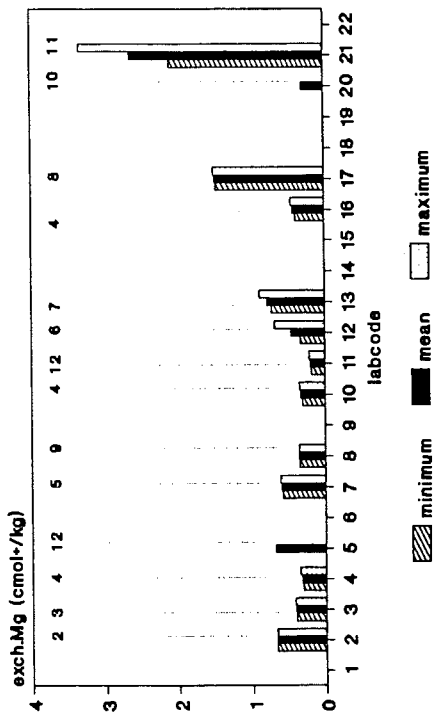
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.352	0.382	0.404	0.026	6.799
5	1		0.950			
6	2	0.630	0.650	0.670	0.028	4.351
7	3	0.710	0.738	0.755	0.025	3.341
8	3	0.330	0.330	0.330	0.000	0.000
9	3	0.630	0.710	0.790	0.080	11.268
10	3	0.860	0.870	0.890	0.017	1.991
11						
12						
13						
14	5	0.768	0.798	0.883	0.048	5.986
15	3	0.710	0.753	0.800	0.045	5.986
16	3	0.790	0.793	0.800	0.006	0.728
17						
18	1		0.425			
19	3		9.000			
20						
21	3	0.640	0.690	0.730	0.046	6.641
22	3	0.586	0.592	0.598	0.006	1.014

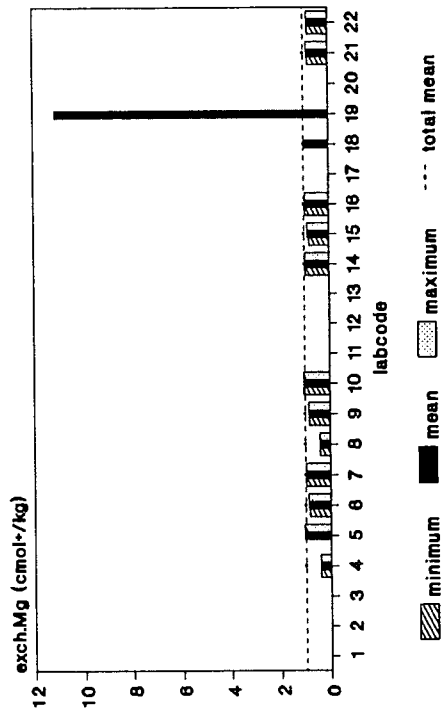
exch.Mg sample B1
national methods



exch.Mg sample B2
national methods



exch.Mg sample B1
reference method



exch.Mg sample B2
reference method

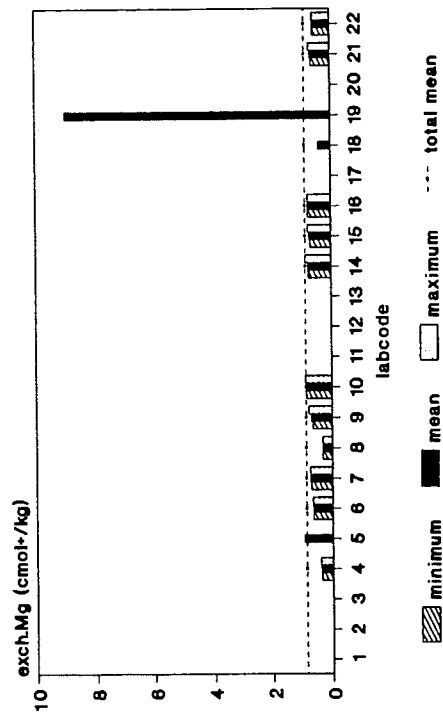


TABLE V : exch.Mg Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 11 2 3 4 7

Number of observations in by group = 27

Dependent Variable: EXCH.Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	43.37530330	0.52	0.7608
Error	21	352.69402944		
Corrected Total	26	396.06933274		

R-Square C.V. EXCH.Mg Mean

0.109514 145.8691 2.80948148

Source	DF	Type I SS	F Value	Pr > F
METH	5	43.37530330	0.52	0.7608

Source	DF	Type III SS	F Value	Pr > F
METH	5	43.37530330	0.52	0.7608

TABLE IV : Exchangeable Mg - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	3.245	5.419	0.382	0.000
A1	2	3	2.237	0.046	0.750	0.000
A1	3	1	1.098			
A1	4	4	0.980	0.007	1.001	0.000
A1	7	3	1.863	0.022	0.986	0.770
A1	11	3	5.450	0.420	1.000	0.992
A2	3	1	3.724			
A2	7	3	5.186	0.164	0.991	0.815
B1	1	38	1.099	1.680	0.259	0.000
B1	2	3	1.047	0.046	0.750	0.000
B1	3	1	0.485			
B1	4	10	0.759	0.297	0.725	0.002
B1	5	3	0.966	0.009	0.999	0.935
B1	6	7	0.604	0.084	0.984	0.974
B1	7	3	0.812	0.009	0.999	0.935
B1	8	3	1.700	0.000	0.000	0.000
B1	9	3	0.413	0.047	0.907	0.407
B1	10	1	0.383	0.479	0.750	0.000
B1	11	3	4.333	0.050	0.878	0.327
B1	12	4	0.920	0.050	0.878	0.327
B2	1	38	0.885	1.364	0.267	0.000
B2	2	3	0.680	0.000	0.000	0.000
B2	3	1	0.418			
B2	4	10	0.365	0.061	0.819	0.024
B2	5	3	0.617	0.020	0.899	0.384
B2	6	6	0.473	0.127	0.902	0.374
B2	7	3	0.792	0.094	0.790	0.092
B2	8	3	1.487	0.015	0.964	0.637
B2	9	3	0.357	0.006	0.750	0.000
B2	10	1	0.296			
B2	11	3	2.650	0.632	0.963	0.631
B2	12	4	0.322	0.252	0.677	0.006

Remark:
Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the
number of reported results.
So, the "repl" values in this table for methods 3 and 1 are different from the real
number of replicates.

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	2	3 7

Number of observations in by group = 4

Dependent Variable: EXCH.Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	1.60381408	59.44	0.0164
Error	2	0.05396867		
Corrected Total	3	1.65778275		
R-Square C.V. EXCH.Mg Mean				
		0.967445	3.407542	4.82075000
Type I SS F Value Pr > F				
Source	DF	Type I SS	F Value	Pr > F
METH	1	1.60381408	59.44	0.0164
Type III SS F Value Pr > F				
Source	DF	Type III SS	F Value	Pr > F
METH	1	1.60381408	59.44	0.0164

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mg

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 21 MSE= 16.79495
Critical Value of Studentized Range= 4.424

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
11 - 1	-6.007		2.205		10.417	
11 - 2	-7.255		3.213		13.682	
11 - 7	-6.881		3.587		14.055	
11 - 3	-10.453		4.352		19.157	
11 - 4	-5.322		4.470		14.262	
1 - 11	-10.417		-2.205		6.007	
1 - 2	-7.203		1.009		9.221	
1 - 7	-6.830		1.382		9.594	
1 - 3	-11.158		2.147		15.452	
1 - 4	-5.065		2.265		9.596	
2 - 11	-13.682		-3.213		7.255	
2 - 1	-9.221		-1.009		7.203	
2 - 7	-10.095		0.374		10.842	
2 - 3	-13.666		1.139		15.943	
2 - 4	-8.536		1.257		11.049	
7 - 11	-14.055		-3.587		6.881	
7 - 1	-9.594		-1.382		6.830	
7 - 2	-10.842		-0.374		10.095	
7 - 3	-14.040		0.765		15.570	
7 - 4	-8.909		0.883		10.675	
3 - 11	-19.157		-4.352		10.453	
3 - 1	-15.452		-2.147		11.158	
3 - 2	-15.943		-1.139		13.666	
3 - 7	-15.570		-0.765		14.040	
3 - 4	-14.216		0.118		14.452	
4 - 11	-14.262		-4.470		5.322	
4 - 1	-9.596		-2.265		5.065	
4 - 2	-11.049		-1.257		8.536	
4 - 7	-10.675		-0.883		8.909	
4 - 3	-14.452		-0.118		14.216	

exch.Mg Method comparison

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 67 MSE= 1.578973

Critical Value of Studentized Range= 4.789

Comparisons significant at the 0.05 level are indicated by '****'.

		Simultaneous		Difference		Simultaneous	
		Lower	Upper	Between	Means	Lower	Upper
		Confidence	Confidence	Means		Limit	Limit
		Limit	Limit				
METH	Comparison						
11	- 8	-0.841	2.633	2.633		6.107	***
11	- 1	0.683	3.235	3.235		5.786	***
11	- 2	-0.187	3.287	3.287		6.761	
11	- 5	-0.107	3.367	3.367		6.841	***
11	- 12	0.164	3.413	3.413		6.663	***
11	- 7	0.048	3.522	3.522		6.996	***
11	- 4	0.774	3.575	3.575		6.375	***
11	- 6	0.793	3.729	3.729		6.665	***
11	- 3	-1.065	3.848	3.848		8.761	***
11	- 9	0.446	3.920	3.920		7.394	***
11	- 10	-0.963	3.950	3.950		8.863	
8	- 11	-6.107	-2.633	-2.633		0.841	
8	- 1	-1.950	0.601	0.601		3.153	
8	- 2	-2.821	0.653	0.653		4.127	
8	- 5	-2.740	0.734	0.734		4.208	
8	- 12	-2.470	0.780	0.780		4.030	
8	- 7	-2.586	0.888	0.888		4.362	
8	- 4	-1.860	0.941	0.941		3.742	
8	- 6	-1.840	1.096	1.096		4.032	
8	- 3	-3.698	1.215	1.215		6.128	
8	- 9	-2.187	1.287	1.287		4.761	
8	- 10	-3.596	1.317	1.317		6.230	
1	- 11	-5.786	-3.235	-3.235		-0.683	***
1	- 8	-3.153	-0.601	-0.601		1.950	
1	- 2	-2.500	0.052	0.052		2.604	
1	- 5	-2.419	0.132	0.132		2.684	
1	- 12	-2.058	0.179	0.179		2.415	
1	- 7	-2.265	0.287	0.287		2.839	
1	- 4	-1.172	0.340	0.340		1.852	
1	- 6	-1.256	0.495	0.495		2.245	
1	- 3	-3.697	0.614	0.614		4.924	
1	- 9	-1.866	0.685	0.685		3.237	
1	- 10	-3.595	0.716	0.716		5.026	
2	- 11	-6.761	-3.287	-3.287		0.187	
2	- 8	-4.127	-0.653	-0.653		2.821	
2	- 1	-2.604	-0.052	-0.052		2.500	

exch.Mg Method comparison

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 12 1 10 11 12 2 3 4 5 6 7 8 9

Number of observations in by group = 79

Dependent Variable: EXCH.Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	38.06386922	2.19	0.0250
Error	67	105.79121068		
Corrected Total	78	143.85507990		
R-Square				
C.V.				
EXCH.Mg Mean				
1.08777215				
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	11	38.06386922	2.19	0.0250
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	11	38.06386922	2.19	0.0250

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit

2	-5	-3.394	0.080	3.554	-0.288	2.513	
2	-12	-3.123	0.127	3.376	-0.208	2.593	
2	-7	-3.239	0.235	3.709	-0.161	2.356	
2	-4	-2.513	0.288	3.089	-0.053	2.748	
2	-6	-2.494	0.442	3.379	0.155	2.251	
2	-3	-4.351	0.562	5.475	0.274	4.736	
2	-9	-2.841	0.633	4.107	0.345	3.146	
2	-10	-4.249	0.664	5.577	0.376	4.838	
5	-11	-6.841	-3.367	0.107	-3.729	-0.793	***
5	-8	-4.208	-0.734	2.740	-1.096	1.840	
5	-1	-2.684	-0.132	2.419	-0.495	1.256	
5	-2	-3.554	-0.080	3.394	-0.442	2.494	
5	-12	-3.203	0.046	3.296	-0.362	2.574	
5	-7	-3.319	0.155	3.629	-0.316	2.351	
5	-4	-2.593	0.208	3.008	-0.207	2.729	
5	-6	-2.574	0.362	3.298	-0.155	1.942	
5	-3	-4.432	0.481	5.394	0.119	4.668	
5	-9	-2.921	0.553	4.027	0.191	3.127	
5	-10	-4.330	0.583	5.496	0.221	4.770	
12	-11	-6.663	-3.413	-0.164	-3.848	1.065	
12	-8	-4.030	-0.780	2.470	-1.215	3.698	
12	-1	-2.415	-0.179	2.058	-0.614	3.697	
12	-2	-3.376	-0.127	3.123	-0.562	4.351	
12	-5	-3.296	-0.046	3.203	-0.481	4.432	
12	-7	-3.141	0.108	3.358	-0.435	4.322	
12	-4	-2.356	0.161	2.678	-0.327	4.586	
12	-6	-2.351	0.316	2.983	-0.274	4.189	
12	-3	-4.322	0.435	5.192	-0.119	4.429	
12	-9	-2.743	0.507	3.756	0.072	4.985	
12	-10	-4.220	0.537	5.294	0.102	6.119	
7	-11	-6.996	-3.522	-0.048	-3.920	-0.446	***
7	-8	-4.362	-0.888	2.586	-1.287	2.187	
7	-1	-2.839	-0.287	2.265	-0.685	1.866	
7	-2	-3.709	-0.235	3.239	-0.633	2.841	
7	-5	-3.629	-0.155	3.319	-0.553	2.921	
7	-12	-3.358	-0.108	3.141	-0.507	2.743	
7	-4	-2.748	0.053	2.854	-0.398	3.076	
7	-6	-2.729	0.207	3.144	-0.345	2.455	
7	-3	-4.586	0.327	5.240	-0.191	2.745	
7	-9	-3.076	0.398	3.872	-0.072	4.841	
7	-10	-4.484	0.429	5.342	0.030	4.943	
4	-11	-6.375	-3.575	-0.774	-3.950	0.963	
4	-8	-3.742	-0.941	1.860	-1.317	3.596	
4	-1	-1.852	-0.340	1.172	-0.716	3.595	

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	12	1 10 11 12 2 3 4 5 6 7 8 9

Number of observations in by group = 78

Dependent Variable: EXCH.Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	16.52869521	1.42	0.1858
Error	66	69.91765294		
Corrected Total	77	86.44634815		

R-Square C.V. EXCH.Mg Mean

0.191202 129.7669 0.79315385

Source	DF	Type I SS	F Value	Pr > F
METH	11	16.52869521	1.42	0.1858
Source	DF	Type III SS	F Value	Pr > F
METH	11	16.52869521	1.42	0.1858

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
10 - 2	-5.577	-0.664	4.249	
10 - 5	-5.496	-0.583	4.330	
10 - 12	-5.294	-0.537	4.220	
10 - 7	-5.342	-0.429	4.484	
10 - 4	-4.838	-0.376	4.087	
10 - 6	-4.770	-0.221	4.327	
10 - 3	-6.119	-0.102	5.915	
10 - 9	-4.943	-0.030	4.883	

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
- 2	-2.736		0.112		2.959
- 5	-2.672		0.175		3.022
- 6	-2.147		0.318		2.784
- 3	-3.653		0.374		4.400
- 4	-1.869		0.426		2.722
- 9	-2.412		0.435		3.282
- 12	-2.194		0.469		3.133
- 10	-3.531		0.496		4.522
- 11	-4.817		-1.970		0.877
- 8	-3.654		-0.807		2.041
- 1	-2.296		-0.205		1.886
- 7	-2.959		-0.112		2.736
- 5	-2.784		0.063		2.911
- 6	-2.259		0.207		2.672
- 3	-3.765		0.262		4.289
- 4	-1.981		0.315		2.610
- 9	-2.524		0.323		3.171
- 12	-2.306		0.358		3.021
- 10	-3.643		0.384		4.411
- 11	-4.881		-2.033		0.814
- 8	-3.717		-0.870		1.977
- 1	-2.359		-0.268		1.823
- 7	-3.022		-0.175		2.672
- 2	-2.911		-0.063		2.784
- 6	-2.322		0.143		2.609
- 3	-3.828		0.199		4.225
- 4	-2.044		0.251		2.547
- 9	-2.587		0.260		3.107
- 12	-2.369		0.294		2.958
- 10	-3.706		0.321		4.347
- 11	-4.642		-2.177		0.289
- 8	-3.479		-1.013		1.452
- 1	-1.943		-0.412		1.120
- 7	-2.784		-0.318		2.147
- 2	-2.672		-0.207		2.259
- 5	-2.609		-0.143		2.322
- 3	-3.711		0.055		3.822
- 4	-1.693		0.108		1.909
- 9	-2.349		0.117		2.582
- 12	-2.100		0.151		2.402
- 10	-3.589		0.177		3.944
- 11	-6.259		-2.232		1.795
- 8	-5.095		-1.069		2.958
- 1	-4.000		-0.467		3.066

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 1.059358
Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
11 - 8	-1.684	1.163	1.163	-1.684	4.011
11 - 1	-0.326	1.765	1.765	-0.326	3.856
11 - 7	-0.989	1.858	1.858	-0.989	4.706
11 - 2	-0.877	1.970	1.970	-0.877	4.817
11 - 5	-0.814	2.033	2.033	-0.814	4.881
11 - 6	-0.289	2.177	2.177	-0.289	4.642
11 - 3	-1.795	2.232	2.232	-1.795	6.259
11 - 4	-0.011	2.285	2.285	-0.011	4.580
11 - 9	-0.554	2.293	2.293	-0.554	5.141
11 - 12	-0.336	2.328	2.328	-0.336	4.991
11 - 10	-1.673	2.354	2.354	-1.673	6.381
8 - 11	-4.011	-1.163	-1.163	-4.011	1.684
8 - 1	-1.489	0.602	0.602	-1.489	2.693
8 - 7	-2.152	0.695	0.695	-2.152	3.542
8 - 2	-2.041	0.807	0.807	-2.041	3.654
8 - 5	-1.977	0.870	0.870	-1.977	3.717
8 - 6	-1.452	1.013	1.013	-1.452	3.479
8 - 3	-2.958	1.069	1.069	-2.958	5.095
8 - 4	-1.174	1.121	1.121	-1.174	3.417
8 - 9	-1.717	1.130	1.130	-1.717	3.977
8 - 12	-1.499	1.164	1.164	-1.499	3.828
8 - 10	-2.836	1.191	1.191	-2.836	5.217
1 - 11	-3.856	-1.765	-1.765	-3.856	0.326
1 - 8	-2.693	-0.602	-0.602	-2.693	1.489
1 - 7	-1.998	0.093	0.093	-1.998	2.184
1 - 2	-1.886	0.205	0.205	-1.886	2.296
1 - 5	-1.823	0.268	0.268	-1.823	2.359
1 - 6	-1.120	0.412	0.412	-1.120	1.943
1 - 3	-3.066	0.467	0.467	-3.066	4.000
1 - 4	-0.720	0.520	0.520	-0.720	1.759
1 - 9	-1.563	0.528	0.528	-1.563	2.619
1 - 12	-1.271	0.562	0.562	-1.271	2.395
1 - 10	-2.944	0.589	0.589	-2.944	4.122
7 - 11	-4.706	-1.858	-1.858	-4.706	0.989
7 - 8	-3.542	-0.695	-0.695	-3.542	2.152
7 - 1	-2.184	-0.093	-0.093	-2.184	1.998

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
10 - 7	-4.522	-0.496	-0.496	-4.522	3.531
10 - 2	-4.411	-0.384	-0.384	-4.411	3.643
10 - 5	-4.347	-0.321	-0.321	-4.347	3.706
10 - 6	-3.944	-0.177	-0.177	-3.944	3.589
10 - 3	-5.054	-0.122	-0.122	-5.054	4.810
10 - 4	-3.727	-0.069	-0.069	-3.727	3.588
10 - 9	-4.087	-0.061	-0.061	-4.087	3.966
10 - 12	-3.925	-0.026	-0.026	-3.925	3.872

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
3 - 7	-4.400	3.653	-0.374	-4.400	3.653
3 - 2	-4.289	3.765	-0.262	-4.289	3.765
3 - 5	-4.225	3.828	-0.199	-4.225	3.828
3 - 6	-3.822	3.711	-0.055	-3.822	3.711
3 - 4	-3.605	3.710	0.053	-3.605	3.710
3 - 9	-3.965	4.088	0.061	-3.965	4.088
3 - 12	-3.803	3.994	0.096	-3.803	3.994
3 - 10	-4.810	5.054	0.122	-4.810	5.054
4 - 11	-4.580	0.011	-2.285	-4.580	0.011
4 - 8	-3.417	1.174	-1.121	-3.417	1.174
4 - 1	-1.759	0.720	-0.520	-1.759	0.720
4 - 7	-2.722	1.869	-0.426	-2.722	1.869
4 - 2	-2.610	1.981	-0.315	-2.610	1.981
4 - 5	-2.547	2.044	-0.251	-2.547	2.044
4 - 6	-1.909	1.693	-0.108	-1.909	1.693
4 - 3	-3.710	3.605	-0.053	-3.710	3.605
4 - 9	-2.287	2.304	0.009	-2.287	2.304
4 - 12	-2.020	2.106	0.043	-2.020	2.106
4 - 10	-3.588	3.727	0.069	-3.588	3.727
9 - 11	-5.141	0.554	-2.293	-5.141	0.554
9 - 8	-3.977	1.717	-1.130	-3.977	1.717
9 - 1	-2.619	1.563	-0.528	-2.619	1.563
9 - 7	-3.282	2.412	-0.435	-3.282	2.412
9 - 2	-3.171	2.524	-0.323	-3.171	2.524
9 - 5	-3.107	2.587	-0.260	-3.107	2.587
9 - 6	-2.582	2.349	-0.117	-2.582	2.349
9 - 3	-4.088	3.965	-0.061	-4.088	3.965
9 - 4	-2.304	2.287	-0.009	-2.304	2.287
9 - 12	-2.629	2.698	0.034	-2.629	2.698
9 - 10	-3.966	4.087	0.061	-3.966	4.087
12 - 11	-4.991	0.336	-2.328	-4.991	0.336
12 - 8	-3.828	1.499	-1.164	-3.828	1.499
12 - 1	-2.395	1.271	-0.562	-2.395	1.271
12 - 7	-3.133	2.194	-0.469	-3.133	2.194
12 - 2	-3.021	2.306	-0.358	-3.021	2.306
12 - 5	-2.958	2.369	-0.294	-2.958	2.369
12 - 6	-2.402	2.100	-0.151	-2.402	2.100
12 - 3	-3.994	3.803	-0.096	-3.994	3.803
12 - 4	-2.106	2.020	-0.043	-2.106	2.020
12 - 9	-2.698	2.629	-0.034	-2.698	2.629
12 - 10	-3.872	3.925	0.026	-3.872	3.925
10 - 11	-6.381	1.673	-2.354	-6.381	1.673
10 - 8	-5.217	2.836	-1.191	-5.217	2.836
10 - 1	-4.122	2.944	-0.589	-4.122	2.944

16. EXCHANGEABLE K

units: cmol+/kg
mandatory for samples B1 and B2
reported by 21 laboratories

TABLE I : Exchangeable K - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1	A1 B1 B2	3	N
2	A1 A2 B1 B2	5	N
3	A1 B1 B2	4	N/R
4	1 not reported		N/R
5	B1 B2	2	R
6	B1 B2	3	N/R
7	B1 B2	3	N/R
8	B1 B2	3	R
9	A1 B1 B2	3	R
10	B1 B2	3	N/R
11	B1 B2	3	N
12	B1 B2	B1:7 B2:6	N
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N
15	B1 B2	3	N=R
16	B1 B2	3	R
17	B1 B2	3	N/R
18	B1 B2	1 not reported	N
19	A1 B1 B2	3	N=R
20	B1 B2	1 not reported	N
21	A1 B1 B2	3	N/R
22	B1 B2	3	R

TABLE IIIa : Exchangeable K - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
2	A1	2	3	0.230	0.237	0.240	0.006	2.440
3	A1	3	5	0.030	0.062	0.100	0.030	48.387
4	A1	4	4	0.154	0.155	0.156	0.001	0.527
13	A1	7	3	0.374	0.392	0.404	0.016	4.007
21	A1	11	3	0.160	0.163	0.170	0.006	3.535
3	A2	3	5	1.720	1.921	2.030	0.150	7.813
13	A2	7	3	2.057	2.144	2.220	0.082	3.825

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	0.340	0.363	0.390	0.025	6.926
2	3	5	0.270	0.276	0.290	0.010	3.623
3	4	4	0.252	0.254	0.255	0.001	0.509
4	12	1		0.270			
6							
7	5	3	0.245	0.248	0.250	0.003	1.067
8	9	3	0.520	0.543	0.570	0.025	4.632
9							
10	4	3	0.240	0.240	0.240	0.000	0.000
11	12	3	0.246	0.250	0.253	0.004	1.442
12	6	7	0.230	0.239	0.250	0.007	2.892
13	7	3	0.339	0.342	0.348	0.005	1.519
14							
15							
16	4	3	0.279	0.284	0.288	0.005	1.590
17	8	3	0.233	0.234	0.235	0.001	0.427
18							
19							
20	10	1		0.169			
21	11	3	0.280	0.287	0.290	0.006	2.014
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	0.180	0.187	0.200	0.012	6.186
3	3	5	0.120	0.123	0.130	0.010	8.130
4	4	4	0.115	0.123	0.130	0.008	6.220
5	12	1		0.120			
6							
7	5	3	0.141	0.147	0.151	0.006	3.738
8	9	3	0.370	0.397	0.410	0.023	5.822
9							
10	4	3	0.050	0.053	0.060	0.006	10.825
11	12	3	0.031	0.032	0.033	0.001	3.646
12	6	6	0.130	0.143	0.150	0.010	7.206
13	7	3	0.190	0.207	0.230	0.021	10.073
14							
15							
16	4	3	0.085	0.100	0.111	0.013	13.361
17	8	3	0.120	0.128	0.132	0.007	5.215
18							
19							
20	10	1		0.115			
21	11	3	0.110	0.137	0.170	0.031	22.354
22							

TABLE II : Exchangeable K - Summary method descriptions

method	description
1	reference, 0.1M BaCl2 1:10
2	1N NH4Ac at pH 7
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
11	1M NH4Ac
12	unspecified

TABLE IIIb : Exchangeable K - descriptive statistics

sample A1 & A2 - reference method

labcode	sample	repl	minimum	mean	maximum	std	cv
4 A1		4	0.160	0.167	0.181	0.010	5.854
14 A1		5	0.147	0.149	0.150	0.001	0.903
19 A1		1	1.000	1.000	1.000		
21 A1		3	0.110	0.123	0.140	0.015	12.385

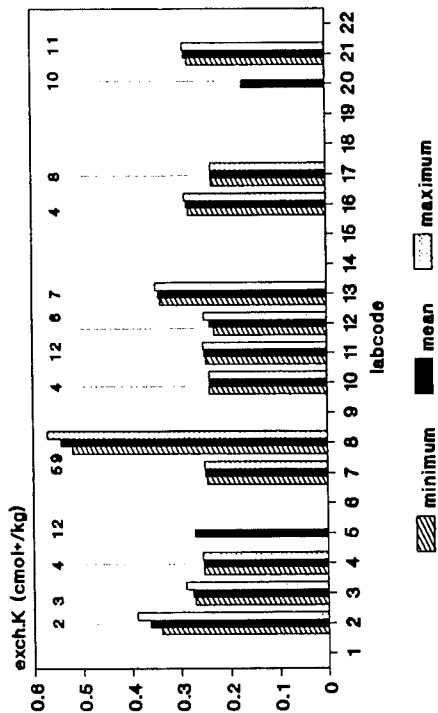
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.198	0.204	0.210	0.005	2.417
5	1		0.210			
6	2	0.18	0.180	0.180	0.000	0.000
7	3	0.209	0.210	0.211	0.001	0.476
8	3	0.23	0.233	0.240	0.006	2.474
9	3	0.21	0.217	0.220	0.006	2.665
10	3	0.18	0.187	0.190	0.006	3.093
11						
12						
13						
14	5	0.182	0.184	0.186	0.002	1.113
15	3	0.19	0.193	0.200	0.006	2.986
16	3	0.183	0.185	0.186	0.002	0.827
17						
18	1		0.284			
19	3		3.000			
20						
21	3	0.16	0.167	0.170	0.006	3.464
22	3	0.21	0.226	0.234	0.014	6.016

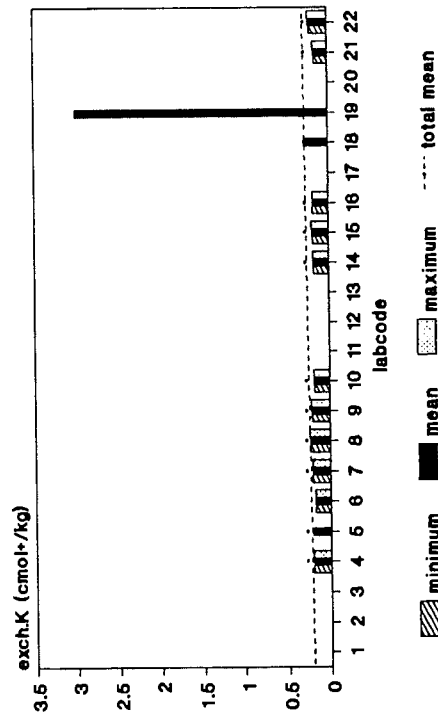
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.112	0.118	0.125	0.007	5.867
5	1	0.14	0.140	0.140		
6	2	0.12	0.120	0.120	0.000	0.000
7	3	0.144	0.153	0.158	0.008	5.105
8	3	0.17	0.173	0.180	0.006	3.331
9	3	0.13	0.137	0.140	0.006	4.225
10	3	0.14	0.140	0.140	0.000	0.000
11						
12						
13						
14	5	0.129	0.132	0.144	0.007	4.912
15	3	0.13	0.137	0.140	0.006	4.225
16	3	0.119	0.122	0.125	0.003	2.511
17						
18	1		0.081			
19	3		1.900			
20						
21	3	0.11	0.130	0.150	0.020	15.385
22	3	0.221	0.226	0.236	0.008	3.705

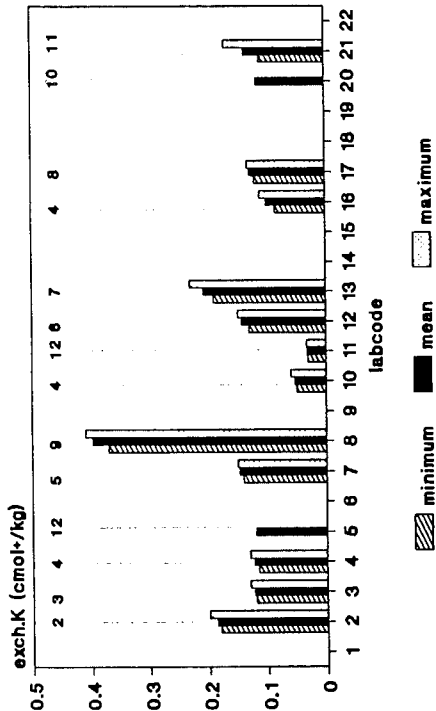
exch.K sample B1
national methods



exch.K sample B1
reference method



exch.K sample B2
national methods



exch.K sample B2
reference method

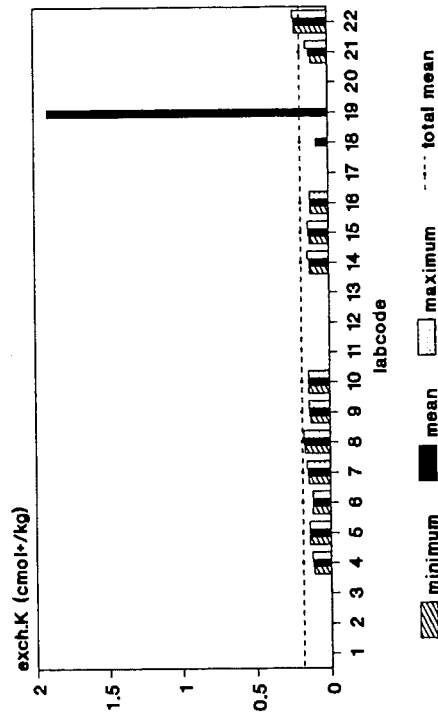


TABLE V : exch.K Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 11 2 3 4 7

Number of observations in by group = 27

Dependent Variable: EXCH.K

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	0.14084310	0.88	0.5131
Error	21	0.67425431		
Corrected Total	26	0.81509741		

R-Square C.V. EXCH.K Mean

0.172793 82.89927 0.21614815

Source	DF	Type I SS	F Value	Pr > F
METH	5	0.14084310	0.88	0.5131
Source	DF	Type III SS	F Value	Pr > F
METH	5	0.14084310	0.88	0.5131

TABLE IV : Exchangeable K - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	0.214	0.237	0.376	0.000
A1	2	3	0.237	0.006	0.750	0.000
A1	3	1	0.062			
A1	4	4	0.155	0.001	0.944	0.654
A1	7	3	0.392	0.016	0.913	0.430
A1	11	3	0.163	0.006	0.750	0.000
A2	3	1	1.921			
A2	7	3	2.144	0.082	0.988	0.791
B1	1	38	0.275	0.455	0.204	0.000
B1	2	3	0.363	0.025	0.987	0.780
B1	3	1	0.276			
B1	4	10	0.258	0.018	0.844	0.047
B1	5	3	0.248	0.003	0.893	0.363
B1	6	7	0.239	0.007	0.841	0.103
B1	7	3	0.342	0.005	0.750	0.000
B1	8	3	0.234	0.001	1.000	0.992
B1	9	3	0.543	0.025	0.987	0.780
B1	10	1	0.169			
B1	11	3	0.287	0.006	0.750	0.000
B1	12	4	0.255	0.010	0.869	0.288
B2	1	38	0.188	0.287	0.242	0.000
B2	2	3	0.187	0.012	0.750	0.000
B2	3	1	0.123			
B2	4	10	0.095	0.032	0.871	0.099
B2	5	3	0.147	0.006	0.824	0.174
B2	6	6	0.143	0.010	0.640	0.000
B2	7	3	0.207	0.021	0.923	0.463
B2	8	3	0.128	0.007	0.812	0.144
B2	9	3	0.397	0.023	0.750	0.000
B2	10	1	0.115			
B2	11	3	0.137	0.031	0.964	0.637
B2	12	4	0.054	0.044	0.646	0.001

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.

To calculate the total number of replicates for the method groups, SAS counts the number of reported results.

So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

exch.K Method comparison

exch.K Method comparison

SAMPLE=A2

SAMPLE=A1

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 21 MSE= 0.032107
Critical Value of Studentized Range= 4.424

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Simultaneous	
	Lower Limit	Difference Between Means	Upper Confidence Limit	
7 - 2	-0.3027	0.1550	0.6127	
7 - 1	-0.1812	0.1779	0.5370	
7 - 11	-0.2294	0.2283	0.6860	
7 - 4	-0.1915	0.2367	0.6648	
7 - 3	-0.3176	0.3297	0.9770	
2 - 7	-0.6127	-0.1550	0.3027	
2 - 1	-0.3362	0.0229	0.3820	
2 - 11	-0.3844	0.0733	0.5310	
2 - 4	-0.3465	0.0817	0.5098	
2 - 3	-0.4726	0.1747	0.8220	
1 - 7	-0.5370	-0.1779	0.1812	
1 - 2	-0.3820	-0.0229	0.3362	
1 - 11	-0.3086	0.0504	0.4095	
1 - 4	-0.2618	0.0588	0.3793	
1 - 3	-0.4300	0.1518	0.7335	
11 - 7	-0.6860	-0.2283	0.2294	
11 - 2	-0.5310	-0.0733	0.3844	
11 - 1	-0.4095	-0.0504	0.3086	
11 - 4	-0.4198	0.0083	0.4365	
11 - 3	-0.5460	0.1013	0.7486	
4 - 7	-0.6648	-0.2367	0.1915	
4 - 2	-0.5098	-0.0817	0.3465	
4 - 1	-0.3793	-0.0588	0.2618	
4 - 11	-0.4365	-0.0083	0.4198	
4 - 3	-0.5337	0.0930	0.7197	
3 - 7	-0.9770	-0.3297	0.3176	
3 - 2	-0.8220	-0.1747	0.4726	
3 - 1	-0.7335	-0.1518	0.4300	
3 - 11	-0.7486	-0.1013	0.5460	
3 - 4	-0.7197	-0.0930	0.5337	

Number of observations in by group = 5

NOTE: Due to missing values, only 4 observations can be used in this analysis.

Dependent Variable: EXCH.K

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	0.03718533	5.53	0.1430
Error	2	0.01344467		
Corrected Total	3	0.05063000		

R-Square C.V. EXCH.K Mean
0.734453 3.926716 2.08800000

Source	DF	Type I SS	F Value	Pr > F
METH	1	0.03718533	5.53	0.1430
Source	DF	Type III SS	F Value	Pr > F
METH	1	0.03718533	5.53	0.1430

Tukey's Studentized Range (HSD) Test for variable: EXCH.K

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of 2 means with 2 error degrees of freedom failed.

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	12	1 10 11 12 2 3 4 5 6 7 8 9

Number of observations in by group = 79

Dependent Variable: EXCH.K

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	0.28235844	0.22	0.9951
Error	67	7.65515908		
Corrected Total	78	7.93751752		
R-Square		C.V.		EXCH.K Mean
	0.035573	120.2153		0.28117722

Source	DF	Type I SS	F Value	Pr > F
METH	11	0.28235844	0.22	0.9951
Source	DF	Type III SS	F Value	Pr > F
METH	11	0.28235844	0.22	0.9951

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 67 MSE= 0.114256
Critical Value of Studentized Range= 4.789

Comparisons significant at the 0.05 level are indicated by '****'.

		Simultaneous		Simultaneous	
		Lower	Difference	Upper	
		Confidence	Between	Confidence	
		Limit	Means	Limit	
METH	Comparison				
9	- 2	-0.7545	0.1800	1.1145	
9	- 7	-0.7332	0.2013	1.1359	
9	- 11	-0.6779	0.2567	1.1912	
9	- 3	-1.0543	0.2673	1.5890	
9	- 1	-0.4178	0.2686	0.9550	
9	- 4	-0.4686	0.2848	1.0383	
9	- 12	-0.5858	0.2883	1.1625	
9	- 5	-0.6392	0.2953	1.2299	
9	- 6	-0.4851	0.3048	1.0946	
9	- 8	-0.6252	0.3093	1.2439	
9	- 10	-0.9473	0.3743	1.6960	
2	- 9	-1.1145	-0.1800	0.7545	
2	- 7	-0.9132	0.0213	0.9559	
2	- 11	-0.8579	0.0767	1.0112	
2	- 3	-1.2343	0.0873	1.4090	
2	- 1	-0.5978	0.0886	0.7750	
2	- 4	-0.6486	0.1048	0.8583	
2	- 12	-0.7658	0.1083	0.9825	
2	- 5	-0.8192	0.1153	1.0499	
2	- 6	-0.6651	0.1248	0.9146	
2	- 8	-0.8052	0.1293	1.0639	
2	- 10	-1.1273	0.1943	1.5160	
7	- 9	-1.1359	-0.2013	0.7332	
7	- 2	-0.9559	-0.0213	0.9132	
7	- 11	-0.8792	0.0553	0.9899	
7	- 3	-1.2556	0.0660	1.3876	
7	- 1	-0.6192	0.0672	0.7536	
7	- 4	-0.6699	0.0835	0.8369	
7	- 12	-0.7872	0.0870	0.9612	
7	- 5	-0.8405	0.0940	1.0285	
7	- 6	-0.6864	0.1034	0.8933	
7	- 8	-0.8265	0.1080	1.0425	
7	- 10	-1.1486	0.1730	1.4946	
11	- 9	-1.1912	-0.2567	0.6779	
11	- 2	-1.0112	-0.0767	0.8579	
11	- 7	-0.9899	-0.0553	0.8792	

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Simultaneous			Simultaneous			Simultaneous			Simultaneous		
METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 3	-1.3110	0.0107	1.3323	12 - 11	-0.9058	-0.0317	0.8425	12 - 11	-0.9058	-0.0317	0.8425
11 - 1	-0.6745	0.0119	0.6983	12 - 3	-1.3007	-0.0210	1.2587	12 - 3	-1.3007	-0.0210	1.2587
11 - 4	-0.7253	0.0282	0.7816	12 - 1	-0.6214	-0.0198	0.5819	12 - 1	-0.6214	-0.0198	0.5819
11 - 12	-0.8425	0.0317	0.9058	12 - 4	-0.6806	-0.0035	0.6736	12 - 4	-0.6806	-0.0035	0.6736
11 - 5	-0.8959	0.0387	0.9732	12 - 5	-0.8672	0.0070	0.8812	12 - 5	-0.8672	0.0070	0.8812
11 - 6	-0.7417	0.0481	0.8379	12 - 6	-0.7010	0.0164	0.7338	12 - 6	-0.7010	0.0164	0.7338
11 - 8	-0.8819	0.0527	0.9872	12 - 8	-0.8532	0.0210	0.8952	12 - 8	-0.8532	0.0210	0.8952
11 - 10	-1.2040	0.1177	1.4393	12 - 10	-1.1937	0.0860	1.3657	12 - 10	-1.1937	0.0860	1.3657
3 - 9	-1.5890	-0.2673	1.0543	5 - 9	-1.2299	-0.2953	0.6392	5 - 9	-1.2299	-0.2953	0.6392
3 - 2	-1.4090	-0.0873	1.2343	5 - 2	-1.0499	-0.1153	0.8192	5 - 2	-1.0499	-0.1153	0.8192
3 - 7	-1.3876	-0.0660	1.2556	5 - 7	-1.0285	-0.0940	0.8405	5 - 7	-1.0285	-0.0940	0.8405
3 - 11	-1.3323	-0.0107	1.3110	5 - 11	-0.9732	-0.0387	0.8959	5 - 11	-0.9732	-0.0387	0.8959
3 - 1	-1.1583	0.0012	1.1608	5 - 3	-1.3496	-0.0280	1.2936	5 - 3	-1.3496	-0.0280	1.2936
3 - 4	-1.1829	0.0175	1.2179	5 - 1	-0.7132	-0.0268	0.6596	5 - 1	-0.7132	-0.0268	0.6596
3 - 12	-1.2587	0.0210	1.3007	5 - 4	-0.7639	-0.0105	0.7429	5 - 4	-0.7639	-0.0105	0.7429
3 - 5	-1.2936	0.0280	1.3496	5 - 12	-0.8812	-0.0070	0.8672	5 - 12	-0.8812	-0.0070	0.8672
3 - 6	-1.1862	0.0374	1.2610	5 - 6	-0.7804	0.0094	0.7993	5 - 6	-0.7804	0.0094	0.7993
3 - 8	-1.2796	0.0420	1.3636	5 - 8	-0.9205	0.0140	0.9485	5 - 8	-0.9205	0.0140	0.9485
3 - 10	-1.5117	0.1070	1.7257	5 - 10	-1.2426	0.0790	1.4006	5 - 10	-1.2426	0.0790	1.4006
1 - 9	-0.9550	-0.2686	0.4178	6 - 9	-1.0946	-0.3048	0.4851	6 - 9	-1.0946	-0.3048	0.4851
1 - 2	-0.7750	-0.0886	0.5978	6 - 2	-0.9146	-0.1248	0.6651	6 - 2	-0.9146	-0.1248	0.6651
1 - 7	-0.7536	-0.0672	0.6192	6 - 7	-0.8933	-0.1034	0.6864	6 - 7	-0.8933	-0.1034	0.6864
1 - 11	-0.6983	-0.0119	0.6745	6 - 11	-0.8379	-0.0481	0.7417	6 - 11	-0.8379	-0.0481	0.7417
1 - 3	-1.1608	-0.0012	1.1583	6 - 3	-1.2610	-0.0374	1.1862	6 - 3	-1.2610	-0.0374	1.1862
1 - 4	-0.3905	0.0163	0.4231	6 - 1	-0.5070	-0.0362	0.4346	6 - 1	-0.5070	-0.0362	0.4346
1 - 12	-0.5819	0.0198	0.6214	6 - 4	-0.5840	-0.0199	0.5441	6 - 4	-0.5840	-0.0199	0.5441
1 - 5	-0.6596	0.0268	0.7132	6 - 12	-0.7338	-0.0164	0.7010	6 - 12	-0.7338	-0.0164	0.7010
1 - 6	-0.4346	0.0362	0.5070	6 - 5	-0.7993	-0.0094	0.7804	6 - 5	-0.7993	-0.0094	0.7804
1 - 8	-0.6456	0.0408	0.7272	6 - 8	-0.7853	0.0046	0.7944	6 - 8	-0.7853	0.0046	0.7944
1 - 10	-1.0538	0.1058	1.2653	6 - 10	-1.1540	0.0696	1.2932	6 - 10	-1.1540	0.0696	1.2932
4 - 9	-1.0383	-0.2848	0.4686	8 - 9	-1.2439	-0.3093	0.6252	8 - 9	-1.2439	-0.3093	0.6252
4 - 2	-0.8583	-0.1048	0.6486	8 - 2	-1.0639	-0.1293	0.8052	8 - 2	-1.0639	-0.1293	0.8052
4 - 7	-0.8369	-0.0835	0.6699	8 - 7	-1.0425	-0.1080	0.8265	8 - 7	-1.0425	-0.1080	0.8265
4 - 11	-0.7816	-0.0282	0.7253	8 - 11	-0.9872	-0.0527	0.8819	8 - 11	-0.9872	-0.0527	0.8819
4 - 3	-1.2179	-0.0175	1.1829	8 - 3	-1.3636	-0.0420	1.2796	8 - 3	-1.3636	-0.0420	1.2796
4 - 1	-0.4231	-0.0163	0.3905	8 - 1	-0.7272	-0.0408	0.6456	8 - 1	-0.7272	-0.0408	0.6456
4 - 12	-0.6736	0.0035	0.6806	8 - 4	-0.7779	-0.0245	0.7289	8 - 4	-0.7779	-0.0245	0.7289
4 - 5	-0.7429	0.0105	0.7639	8 - 12	-0.8952	-0.0210	0.8532	8 - 12	-0.8952	-0.0210	0.8532
4 - 6	-0.5441	0.0199	0.5840	8 - 5	-0.9485	-0.0140	0.9205	8 - 5	-0.9485	-0.0140	0.9205
4 - 8	-0.7289	0.0245	0.7779	8 - 6	-0.7944	-0.0046	0.7853	8 - 6	-0.7944	-0.0046	0.7853
4 - 10	-1.1109	0.0895	1.2899	8 - 10	-1.2566	0.0650	1.3866	8 - 10	-1.2566	0.0650	1.3866
12 - 9	-1.1625	-0.2883	0.5858	10 - 9	-1.6960	-0.3743	0.9473	10 - 9	-1.6960	-0.3743	0.9473
12 - 2	-0.9825	-0.1083	0.7658	10 - 2	-1.5160	-0.1943	1.1273	10 - 2	-1.5160	-0.1943	1.1273
12 - 7	-0.9612	-0.0870	0.7872	10 - 7	-1.4946	-0.1730	1.1486	10 - 7	-1.4946	-0.1730	1.1486

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	12	1 10 11 12 2 3 4 5 6 7 8 9

Number of observations in by group = 78

Dependent Variable: EXCH.K

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	0.30040544	0.59	0.8318
Error	66	3.06478524		
Corrected Total	77	3.36519068		
R-Square				
			C.V.	EXCH.K Mean
			129.0858	0.16693590

Source	DF	Type I SS	F Value	Pr > F
METH	11	0.30040544	0.59	0.8318
Source	DF	Type III SS	F Value	Pr > F
METH	11	0.30040544	0.59	0.8318

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	Lower	Upper			Lower	Upper
10 - 11	-1.4393	1.2040	-0.1177		1.5117	
10 - 3	-1.7257	1.5117	-0.1070		1.0538	
10 - 1	-1.2653	1.0538	-0.1058		1.1109	
10 - 4	-1.2899	1.1109	-0.0895		1.1937	
10 - 12	-1.3657	1.1937	-0.0860		1.2426	
10 - 5	-1.4006	1.2426	-0.0790		1.1540	
10 - 6	-1.2932	1.1540	-0.0696		1.2566	
10 - 8	-1.3866	1.2566	-0.0650			

----- SAMPLE=B2 -----

----- SAMPLE=B2 -----

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 66 MSE= 0.046436
Critical Value of Studentized Range= 4.791

Comparisons significant at the 0.05 level are indicated by '****'.

Simultaneous

Lower Confidence Limit
Difference Between Means
Upper Confidence Limit

METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
9 - 7	-0.4061	0.1900	0.7861
9 - 1	-0.2293	0.2085	0.6463
9 - 2	-0.3861	0.2100	0.8061
9 - 5	-0.3468	0.2493	0.8455
9 - 6	-0.2629	0.2533	0.7696
9 - 11	-0.3361	0.2600	0.8561
9 - 8	-0.3271	0.2690	0.8651
9 - 3	-0.5694	0.2737	1.1167
9 - 10	-0.5614	0.2817	1.1247
9 - 4	-0.1792	0.3014	0.7820
9 - 12	-0.2147	0.3429	0.9005

7 - 9	-0.7861	-0.1900	0.4061
7 - 1	-0.4193	0.0185	0.4563
7 - 2	-0.5761	0.0200	0.6161
7 - 5	-0.5368	0.0593	0.6555
7 - 6	-0.4529	0.0633	0.5796
7 - 11	-0.5261	0.0700	0.6661
7 - 8	-0.5171	0.0790	0.6751
7 - 3	-0.7594	0.0837	0.9267
7 - 10	-0.7514	0.0917	0.9347
7 - 4	-0.3692	0.1114	0.5920
7 - 12	-0.4047	0.1529	0.7105

1 - 9	-0.6463	-0.2085	0.2293
1 - 7	-0.4563	-0.0185	0.4193
1 - 2	-0.4363	0.0015	0.4393
1 - 5	-0.3970	0.0408	0.4787
1 - 6	-0.2759	0.0448	0.3656
1 - 11	-0.3863	0.0515	0.4893
1 - 8	-0.3773	0.0605	0.4983
1 - 3	-0.6745	0.0652	0.8048
1 - 10	-0.6665	0.0732	0.8128
1 - 4	-0.1666	0.0929	0.3523
1 - 12	-0.2494	0.1344	0.5182

2 - 9	-0.8061	-0.2100	0.3861
2 - 7	-0.6161	-0.0200	0.5761
2 - 1	-0.4393	-0.0015	0.4363

Simultaneous

Lower Confidence Limit
Difference Between Means
Upper Confidence Limit

2 - 5	-0.5568	0.0393	0.6355
2 - 6	-0.4729	0.0433	0.5596
2 - 11	-0.5461	0.0500	0.6461
2 - 8	-0.5371	0.0590	0.6551
2 - 3	-0.7794	0.0637	0.9067
2 - 10	-0.7714	0.0717	0.9147
2 - 4	-0.3892	0.0914	0.5720
2 - 12	-0.4247	0.1329	0.6905

5 - 9	-0.8455	-0.2493	0.3468
5 - 7	-0.6555	-0.0593	0.5368
5 - 1	-0.4787	-0.0408	0.3970
5 - 2	-0.6355	-0.0393	0.5568
5 - 6	-0.5123	0.0040	0.5203
5 - 11	-0.5855	0.0107	0.6068
5 - 8	-0.5765	0.0197	0.6158
5 - 3	-0.8187	0.0243	0.8674
5 - 10	-0.8107	0.0323	0.8754
5 - 4	-0.4286	0.0520	0.5326
5 - 12	-0.4640	0.0936	0.6512

6 - 9	-0.7696	-0.2533	0.2629
6 - 7	-0.5796	-0.0633	0.4529
6 - 1	-0.3656	-0.0448	0.2759
6 - 2	-0.5596	-0.0433	0.4729
6 - 5	-0.5203	-0.0040	0.5123
6 - 11	-0.5096	0.0067	0.5229
6 - 8	-0.5006	0.0157	0.5319
6 - 3	-0.7683	0.0203	0.8089
6 - 10	-0.7603	0.0283	0.8169
6 - 4	-0.3290	0.0480	0.4251
6 - 12	-0.3817	0.0896	0.5609

11 - 9	-0.8561	-0.2600	0.3361
11 - 7	-0.6661	-0.0700	0.5261
11 - 1	-0.4893	-0.0515	0.3863
11 - 2	-0.6461	-0.0500	0.5461
11 - 5	-0.6068	-0.0107	0.5855
11 - 6	-0.5229	-0.0067	0.5096
11 - 8	-0.5871	0.0090	0.6051
11 - 3	-0.8294	0.0137	0.8567
11 - 10	-0.8214	0.0217	0.8647
11 - 4	-0.4392	0.0414	0.5220
11 - 12	-0.4747	0.0829	0.6405

8 - 9	-0.8651	-0.2690	0.3271
8 - 7	-0.6751	-0.0790	0.5171
8 - 1	-0.4983	-0.0605	0.3773

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	Limit					
12 - 2	-0.6905		-0.1329		0.4247	
12 - 5	-0.6512		-0.0936		0.4640	
12 - 6	-0.5609		-0.0896		0.3817	
12 - 11	-0.6405		-0.0829		0.4747	
12 - 8	-0.6315		-0.0739		0.4837	
12 - 3	-0.8855		-0.0693		0.7470	
12 - 10	-0.8775		-0.0613		0.7550	
12 - 4	-0.4735		-0.0416		0.3904	

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	Limit					
8 - 2	-0.6551		-0.0590		0.5371	
8 - 5	-0.6158		-0.0197		0.5765	
8 - 6	-0.5319		-0.0157		0.5006	
8 - 11	-0.6051		-0.0090		0.5871	
8 - 3	-0.8384		0.0047		0.8477	
8 - 10	-0.8304		0.0127		0.8557	
8 - 4	-0.4482		0.0324		0.5130	
8 - 12	-0.4837		0.0739		0.6315	
3 - 9	-1.1167		-0.2737		0.5694	
3 - 7	-0.9267		-0.0837		0.7594	
3 - 1	-0.8048		-0.0652		0.6745	
3 - 2	-0.9067		-0.0637		0.7794	
3 - 5	-0.8674		-0.0243		0.8187	
3 - 6	-0.8089		-0.0203		0.7683	
3 - 11	-0.8567		-0.0137		0.8294	
3 - 8	-0.8477		-0.0047		0.8384	
3 - 10	-1.0245		0.0080		1.0405	
3 - 4	-0.7380		0.0277		0.7934	
3 - 12	-0.7470		0.0693		0.8855	
10 - 9	-1.1247		-0.2817		0.5614	
10 - 7	-0.9347		-0.0917		0.7514	
10 - 1	-0.8128		-0.0732		0.6665	
10 - 2	-0.9147		-0.0717		0.7714	
10 - 5	-0.8754		-0.0323		0.8107	
10 - 6	-0.8169		-0.0283		0.7603	
10 - 11	-0.8647		-0.0217		0.8214	
10 - 8	-0.8557		-0.0127		0.8304	
10 - 3	-1.0405		-0.0080		1.0245	
10 - 4	-0.7460		0.0197		0.7854	
10 - 12	-0.7550		0.0613		0.8775	
4 - 9	-0.7820		-0.3014		0.1792	
4 - 7	-0.5920		-0.1114		0.3692	
4 - 1	-0.3523		-0.0929		0.1666	
4 - 2	-0.5720		-0.0914		0.3892	
4 - 5	-0.5326		-0.0520		0.4286	
4 - 6	-0.4251		-0.0480		0.3290	
4 - 11	-0.5220		-0.0414		0.4392	
4 - 8	-0.5130		-0.0324		0.4482	
4 - 3	-0.7934		-0.0277		0.7380	
4 - 10	-0.7854		-0.0197		0.7460	
4 - 12	-0.3904		0.0416		0.4735	
12 - 9	-0.9005		-0.3429		0.2147	
12 - 7	-0.7105		-0.1529		0.4047	
12 - 1	-0.5182		-0.1344		0.2494	

17. EXCHANGEABLE Na

units: cmol+/kg
mandatory for samples B1 and B2
reported by 18 laboratories

TABLE I : Exchangeable Na - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2			
3	A1 A2 B1 B2	5	N
4	A1 B1 B2	4	N/R
5	B1 B2	1 not reported	N/R
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9			
10	B1 B2	3	N/R
11	B1 B2	3	N
12	B1 B2	B1:7 B2:6	N
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N=R
15	B1 B2	3	R
16	B1 B2	3	N/R
17			
18	B1 B2	1 not reported	N=R
19	A1 B1 B2	3	N=R
20	B1 B2	1 not reported	N
21	A1 B1 B2	3	N/R
22	B1 B2	3	R

TABLE II : Exchangeable Na - Summary method description

method	description
1	reference
2	1N NH4Ac at pH 7
3	1M NH4NO3
4	1M NH4Cl
5	AFNOR X31-130
6	0.2N BaCl2 at pH 8.1
7	0.01M AgTU
8	1M KCl at pH 8.2, next: 4% NaF at pH 8.2
9	1N CH3COONH4 at pH 7
10	0.1M BaCl2
11	1M NH4Ac
12	unspecified

* not applicable

TABLE IIIa : Exchangeable Na - descriptive statistics

sample A1 & A2 - national methods

labcode	sample	method	repl	minimum	mean	maximum	std	cv
3	A1	3	5	0.085	0.091	0.097	0.005	5.495
4	A1	4	4	0.071	0.072	0.074	0.001	1.742
13	A1	7	3	0.058	0.081	0.098	0.021	25.446
21	A1	11	3	0.180	0.193	0.210	0.015	7.901
3	A2	3	5	0.101	0.103	0.106	0.002	1.942
13	A2	7	3	0.003	0.033	0.085	0.045	137.002

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.052	0.053	0.055	0.001	1.887
4	4	4	0.047	0.048	0.050	0.001	2.608
5	12	1		0.060			
6							
7	5	3	0.066	0.068	0.069	0.002	2.257
8	9	3	0.360	0.380	0.390	0.017	4.558
9							
10	4	3	0.060	0.063	0.070	0.006	9.116
11	12	3	0.048	0.050	0.052	0.002	4.136
12	6	7	0.090	0.161	0.290	0.083	51.386
13	7	3	0.041	0.046	0.055	0.008	16.342
14							
15							
16	4	3	0.058	0.063	0.071	0.007	10.748
17							
18							
19							
20	10	1		0.022			
21	11	3	0.090	0.103	0.120	0.015	14.783
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	3	5	0.038	0.041	0.043	0.002	4.878
4	4	4	0.035	0.040	0.041	0.003	7.595
5	12	1		0.040			
6							
7	5	3	0.051	0.058	0.068	0.009	15.735
8	9	3	0.540	0.570	0.600	0.030	5.263
9							
10	4	3	0.030	0.033	0.040	0.006	17.321
11	12	3	0.016	0.017	0.017	0.001	3.464
12	6	6	0.110	0.217	0.300	0.086	39.881
13	7	3	0.031	0.037	0.045	0.007	20.103
14							
15							
16	4	3	0.037	0.049	0.067	0.016	32.397
17							
18							
19							
20	10	1		0.017			
21	11	3	0.150	0.173	0.210	0.032	18.545
22							

TABLE IIIb : Exchangeable Na - descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
4	4	0.069	0.075	0.078	0.004	5.393
14	5	0.068	0.069	0.070	0.001	1.209
19	3		0.310			
21	3	0.090	0.090	0.090	0.000	0.000

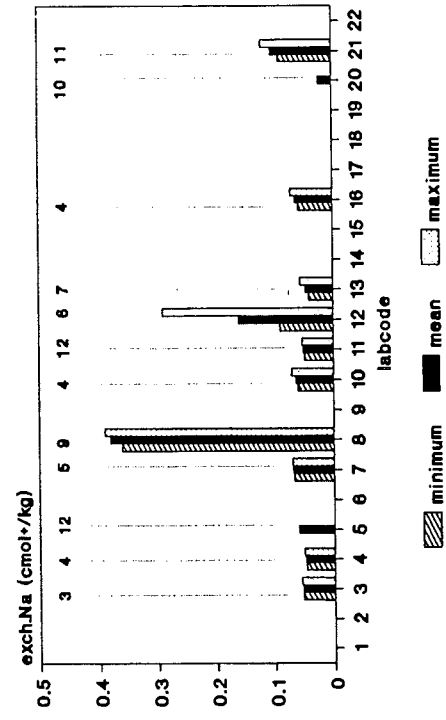
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.050	0.051	0.053	0.001	2.773
5	1		0.080			
6	2	0.040	0.040	0.040	0.000	0.000
7	3	0.059	0.061	0.063	0.002	3.431
8	3	0.040	0.300	0.430	0.225	75.056
9						
10	3	0.060	0.063	0.070	0.006	9.116
11						
12						
13						
14	5	0.045	0.046	0.047	0.001	2.174
15	3	0.049	0.049	0.053	0.003	5.771
16	3	0.044	0.050	0.058	0.007	14.841
17						
18	1		0.213			
19	3		0.200			
20						
21	3	0.060	0.067	0.070	0.006	8.660
22	3	0.113	0.119	0.126	0.007	5.510

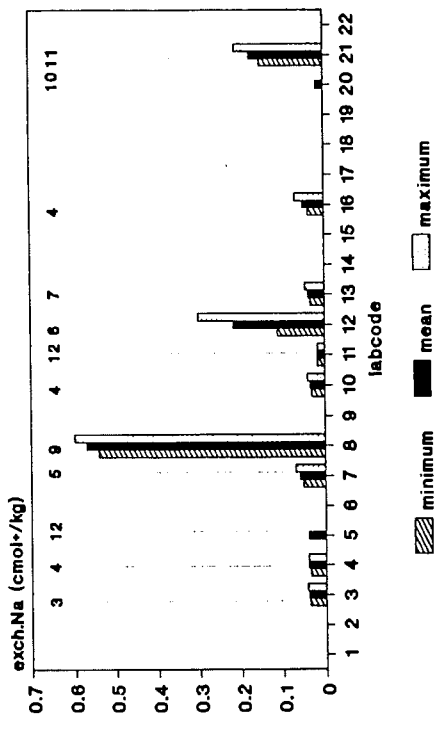
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	0.039	0.041	0.044	0.002	4.998
5	1		0.070			
6	2	0.030	0.030	0.030	0.000	0.000
7	3	0.048	0.051	0.053	0.003	5.624
8	3	0.390	0.397	0.400	0.006	1.456
9						
10	3	0.060	0.067	0.070	0.006	8.660
11						
12						
13						
14	5	0.035	0.037	0.040	0.002	5.227
15	3	0.035	0.039	0.047	0.007	17.692
16	3	0.026	0.039	0.052	0.013	33.083
17						
18	1		0.172			
19	1		0.200			
20						
21	3	0.040	0.047	0.050	0.006	12.372
22	3	0.095	0.096	0.098	0.002	1.586

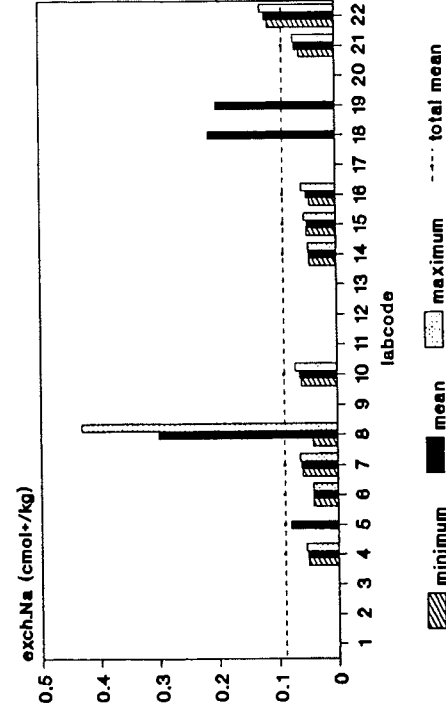
exch.Na sample B1
national methods



exch.Na sample B2
national methods



exch.Na sample B1
reference method



exch.Na sample B2
reference method

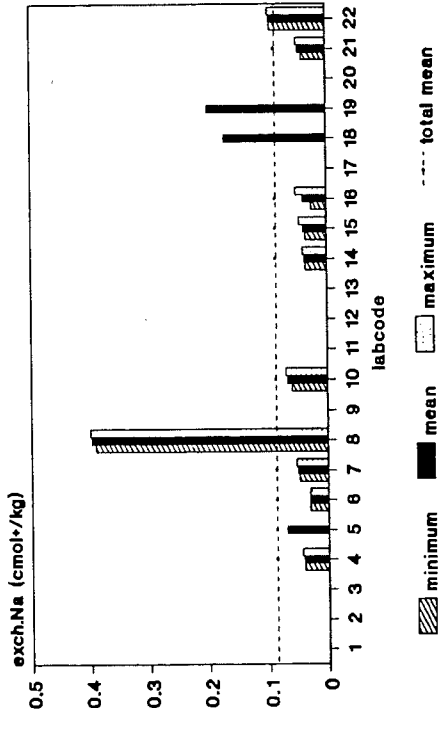


TABLE V : exch.Na Method comparison

----- SAMPLE=Al -----

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 5 1 11 3 4 7

Number of observations in by group = 24

Dependent Variable: EXCH.Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	0.03081823	2.78	0.0565
Error	19	0.05262639		
Corrected Total	23	0.08344463		

R-Square C.V. EXCH.Na Mean

0.369326 52.04350 0.10112500

Source	DF	Type I SS	F Value	Pr > F
METH	4	0.03081823	2.78	0.0565
Source	DF	Type III SS	F Value	Pr > F
METH	4	0.03081823	2.78	0.0565

TABLE IV : Exchangeable Na - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	0.094	0.065	0.414	0.000
A1	3	1	0.091			
A1	4	4	0.072	0.001	0.895	0.400
A1	7	3	0.081	0.021	0.949	0.567
A1	11	3	0.193	0.015	0.964	0.637
A2	3	1	0.103			
A2	7	3	0.033	0.045	0.822	0.169
B1	1	35	0.090	0.094	0.524	0.000
B1	3	1	0.053			
B1	4	10	0.057	0.009	0.891	0.168
B1	5	3	0.068	0.002	0.964	0.637
B1	6	7	0.161	0.083	0.785	0.029
B1	7	3	0.046	0.008	0.855	0.253
B1	9	3	0.380	0.017	0.750	0.000
B1	10	1	0.022			
B1	11	3	0.103	0.015	0.964	0.637
B1	12	4	0.053	0.005	0.899	0.420
B2	1	35	0.087	0.103	0.554	0.000
B2	3	1	0.041			
B2	4	10	0.040	0.010	0.763	0.006
B2	5	3	0.058	0.009	0.878	0.317
B2	6	6	0.217	0.086	0.838	0.117
B2	7	3	0.037	0.007	0.902	0.391
B2	9	3	0.570	0.030	1.000	0.992
B2	10	1	0.017			
B2	11	3	0.173	0.032	0.871	0.298
B2	12	4	0.023	0.012	0.665	0.004

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.

So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

----- SAMPLE=A2 -----

General Linear Models Procedure
Class Level Information

Class Levels Values
METH 2 3 7

Number of observations in by group = 4

Dependent Variable: EXCH.Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	0.00367500	1.80	0.3120
Error	2	0.00408800		
Corrected Total	3	0.00776300		

R-Square C.V. EXCH.Na Mean
0.473399 89.52598 0.05050000

Source	DF	Type I SS	F Value	Pr > F
METH	1	0.00367500	1.80	0.3120
Source	DF	Type III SS	F Value	Pr > F
METH	1	0.00367500	1.80	0.3120

Tukey's Studentized Range (HSD) Test for variable: EXCH.Na

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

----- SAMPLE=A1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 19 MSE= 0.00277
Critical Value of Studentized Range= 4.253

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
11 - 1	-0.0023	0.0991	0.0991	0.2005	0.2005	0.2005
11 - 3	-0.0804	0.1023	0.1023	0.2851	0.2851	0.2851
11 - 7	-0.0166	0.1127	0.1127	0.2419	0.2419	0.2419
11 - 4	0.0002	0.1211	0.1211	0.2420	0.2420	***
1 - 11	-0.2005	-0.0991	-0.0991	0.0023	0.0023	0.0023
1 - 3	-0.1610	0.0032	0.0032	0.1675	0.1675	0.1675
1 - 7	-0.0878	0.0136	0.0136	0.1149	0.1149	0.1149
1 - 4	-0.0685	0.0220	0.0220	0.1125	0.1125	0.1125
3 - 11	-0.2851	-0.1023	-0.1023	0.0804	0.0804	0.0804
3 - 1	-0.1675	-0.0032	-0.0032	0.1610	0.1610	0.1610
3 - 7	-0.1724	0.0103	0.0103	0.1931	0.1931	0.1931
3 - 4	-0.1582	0.0188	0.0188	0.1957	0.1957	0.1957
7 - 11	-0.2419	-0.1127	-0.1127	0.0166	0.0166	0.0166
7 - 1	-0.1149	-0.0136	-0.0136	0.0878	0.0878	0.0878
7 - 3	-0.1931	-0.0103	-0.0103	0.1724	0.1724	0.1724
7 - 4	-0.1125	0.0084	0.0084	0.1293	0.1293	0.1293
4 - 11	-0.2420	-0.1211	-0.1211	-0.0002	-0.0002	***
4 - 1	-0.1125	-0.0220	-0.0220	0.0685	0.0685	0.0685
4 - 3	-0.1957	-0.0188	-0.0188	0.1582	0.1582	0.1582
4 - 7	-0.1293	-0.0084	-0.0084	0.1125	0.1125	0.1125

----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 12 3 4 5 6 7 9

Number of observations in by group = 70

Dependent Variable: EXCH.Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	0.31256762	6.06	0.0001
Error	60	0.34387696		
Corrected Total	69	0.65644459		
R-Square		C.V.		EXCH.Na Mean
0.476152		76.61365		0.09881429

Source	DF	Type I SS	F Value	Pr > F
METH	9	0.31256762	6.06	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	0.31256762	6.06	0.0001

----- SAMPLE=B1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 60 MSE= 0.005731
Critical Value of Studentized Range= 4.646

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
9 - 6	0.0469	0.2186	0.2186	0.0469	0.3902
9 - 11	0.0736	0.2767	0.2767	0.0736	0.4798
9 - 1	0.1408	0.2904	0.2904	0.1408	0.4400
9 - 5	0.1092	0.3123	0.3123	0.1092	0.5154
9 - 4	0.1590	0.3227	0.3227	0.1590	0.4864
9 - 3	0.0398	0.3270	0.3270	0.0398	0.6142
9 - 12	0.1373	0.3273	0.3273	0.1373	0.5172
9 - 7	0.1306	0.3337	0.3337	0.1306	0.5368
9 - 10	0.0708	0.3580	0.3580	0.0708	0.6452
6 - 9	-0.3902	-0.2186	-0.2186	-0.3902	-0.0469
6 - 11	-0.1135	0.0581	0.0581	-0.1135	0.2297
6 - 1	-0.0312	0.0718	0.0718	-0.0312	0.1748
6 - 5	-0.0779	0.0938	0.0938	-0.0779	0.2654
6 - 4	-0.0184	0.1041	0.1041	-0.0184	0.2267
6 - 3	-0.1575	0.1084	0.1084	-0.1575	0.3743
6 - 12	-0.0472	0.1087	0.1087	-0.0472	0.2646
6 - 7	-0.0565	0.1151	0.1151	-0.0565	0.2867
6 - 10	-0.1265	0.1394	0.1394	-0.1265	0.4053
11 - 9	-0.4798	-0.2767	-0.2767	-0.4798	-0.0736
11 - 6	-0.2297	-0.0581	-0.0581	-0.2297	0.1135
11 - 1	-0.1359	0.0137	0.0137	-0.1359	0.1634
11 - 5	-0.1674	0.0357	0.0357	-0.1674	0.2388
11 - 4	-0.1177	0.0460	0.0460	-0.1177	0.2098
11 - 3	-0.2369	0.0503	0.0503	-0.2369	0.3375
11 - 12	-0.1394	0.0506	0.0506	-0.1394	0.2406
11 - 7	-0.1461	0.0570	0.0570	-0.1461	0.2601
11 - 10	-0.2059	0.0813	0.0813	-0.2059	0.3685
1 - 9	-0.4400	-0.2904	-0.2904	-0.4400	-0.1408
1 - 6	-0.1748	-0.0718	-0.0718	-0.1748	0.0312
1 - 11	-0.1634	-0.0137	-0.0137	-0.1634	0.1359
1 - 5	-0.1277	0.0219	0.0219	-0.1277	0.1716
1 - 4	-0.0569	0.0323	0.0323	-0.0569	0.1215
1 - 3	-0.2157	0.0366	0.0366	-0.2157	0.2889
1 - 12	-0.0944	0.0368	0.0368	-0.0944	0.1681
1 - 7	-0.1064	0.0433	0.0433	-0.1064	0.1929
1 - 10	-0.1847	0.0676	0.0676	-0.1847	0.3199

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
7 - 10	-0.2629	0.0243	0.3115	-0.2629	0.0243	0.3115
10 - 9	-0.6452	-0.3580	-0.0708	-0.6452	-0.3580	-0.0708
10 - 6	-0.4053	-0.1394	0.1265	-0.4053	-0.1394	0.1265
10 - 11	-0.3685	-0.0813	0.2059	-0.3685	-0.0813	0.2059
10 - 1	-0.3199	-0.0676	0.1847	-0.3199	-0.0676	0.1847
10 - 5	-0.3329	-0.0457	0.2415	-0.3329	-0.0457	0.2415
10 - 4	-0.2962	-0.0353	0.2256	-0.2962	-0.0353	0.2256
10 - 3	-0.3828	-0.0310	0.3208	-0.3828	-0.0310	0.3208
10 - 12	-0.3088	-0.0307	0.2473	-0.3088	-0.0307	0.2473
10 - 7	-0.3115	-0.0243	0.2629	-0.3115	-0.0243	0.2629

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
5 - 9	-0.5154	-0.3123	-0.1092	-0.5154	-0.3123	-0.1092
5 - 6	-0.2654	-0.0938	0.0779	-0.2654	-0.0938	0.0779
5 - 11	-0.2388	-0.0357	0.1674	-0.2388	-0.0357	0.1674
5 - 1	-0.1716	-0.0219	0.1277	-0.1716	-0.0219	0.1277
5 - 4	-0.1534	0.0104	0.1741	-0.1534	0.0104	0.1741
5 - 3	-0.2725	0.0147	0.3019	-0.2725	0.0147	0.3019
5 - 12	-0.1751	0.0149	0.2049	-0.1751	0.0149	0.2049
5 - 7	-0.1818	0.0213	0.2244	-0.1818	0.0213	0.2244
5 - 10	-0.2415	0.0457	0.3329	-0.2415	0.0457	0.3329
4 - 9	-0.4864	-0.3227	-0.1590	-0.4864	-0.3227	-0.1590
4 - 6	-0.2267	-0.1041	0.0184	-0.2267	-0.1041	0.0184
4 - 11	-0.2098	-0.0460	0.1177	-0.2098	-0.0460	0.1177
4 - 1	-0.1215	-0.0323	0.0569	-0.1215	-0.0323	0.0569
4 - 5	-0.1741	-0.0104	0.1534	-0.1741	-0.0104	0.1534
4 - 3	-0.2566	0.0043	0.2652	-0.2566	0.0043	0.2652
4 - 12	-0.1426	0.0046	0.1517	-0.1426	0.0046	0.1517
4 - 7	-0.1528	0.0110	0.1747	-0.1528	0.0110	0.1747
4 - 10	-0.2256	0.0353	0.2962	-0.2256	0.0353	0.2962
3 - 9	-0.6142	-0.3270	-0.0398	-0.6142	-0.3270	-0.0398
3 - 6	-0.3743	-0.1084	0.1575	-0.3743	-0.1084	0.1575
3 - 11	-0.3375	-0.0503	0.2369	-0.3375	-0.0503	0.2369
3 - 1	-0.2889	-0.0366	0.2157	-0.2889	-0.0366	0.2157
3 - 5	-0.3019	-0.0147	0.2725	-0.3019	-0.0147	0.2725
3 - 4	-0.2652	-0.0043	0.2566	-0.2652	-0.0043	0.2566
3 - 12	-0.2778	0.0003	0.2783	-0.2778	0.0003	0.2783
3 - 7	-0.2805	0.0067	0.2939	-0.2805	0.0067	0.2939
3 - 10	-0.3208	0.0310	0.3828	-0.3208	0.0310	0.3828
12 - 9	-0.5172	-0.3273	-0.1373	-0.5172	-0.3273	-0.1373
12 - 6	-0.2646	-0.1087	0.0472	-0.2646	-0.1087	0.0472
12 - 11	-0.2406	-0.0506	0.1394	-0.2406	-0.0506	0.1394
12 - 1	-0.1681	-0.0368	0.0944	-0.1681	-0.0368	0.0944
12 - 5	-0.2049	-0.0149	0.1751	-0.2049	-0.0149	0.1751
12 - 4	-0.1517	-0.0046	0.1426	-0.1517	-0.0046	0.1426
12 - 3	-0.2783	-0.0003	0.2778	-0.2783	-0.0003	0.2778
12 - 7	-0.1836	0.0064	0.1964	-0.1836	0.0064	0.1964
12 - 10	-0.2473	0.0307	0.3088	-0.2473	0.0307	0.3088
7 - 9	-0.5368	-0.3337	-0.1306	-0.5368	-0.3337	-0.1306
7 - 6	-0.2867	-0.1151	0.0565	-0.2867	-0.1151	0.0565
7 - 11	-0.2601	-0.0570	0.1461	-0.2601	-0.0570	0.1461
7 - 1	-0.1929	-0.0433	0.1064	-0.1929	-0.0433	0.1064
7 - 5	-0.2244	-0.0213	0.1818	-0.2244	-0.0213	0.1818
7 - 4	-0.1747	-0.0110	0.1528	-0.1747	-0.0110	0.1528
7 - 3	-0.2939	-0.0067	0.2805	-0.2939	-0.0067	0.2805
7 - 12	-0.1964	-0.0064	0.1836	-0.1964	-0.0064	0.1836

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 11 12 3 4 5 6 7 9

Number of observations in by group = 69

Dependent Variable: EXCH.Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	0.84926920	13.82	0.0001
Error	59	0.40289323		
Corrected Total	68	1.25216243		
R-Square		C.V.	EXCH.Na Mean	
0.678242		76.79300	0.10760870	

Source	DF	Type I SS	F Value	Pr > F
METH	9	0.84926920	13.82	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	0.84926920	13.82	0.0001

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: EXCH.Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 59 MSE= 0.006829
Critical Value of Studentized Range= 4.649

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
9 - 6	0.1612	0.3533	0.3533	0.1612	0.5454
9 - 11	0.1748	0.3967	0.3967	0.1748	0.6185
9 - 1	0.3192	0.4826	0.4826	0.3192	0.6460
9 - 5	0.2905	0.5123	0.5123	0.2905	0.7342
9 - 3	0.2153	0.5290	0.5290	0.2153	0.8427
9 - 4	0.3507	0.5295	0.5295	0.3507	0.7083
9 - 7	0.3115	0.5333	0.5333	0.3115	0.7552
9 - 12	0.3400	0.5475	0.5475	0.3400	0.7550
9 - 10	0.2393	0.5530	0.5530	0.2393	0.8667
6 - 9	-0.5454	-0.3533	-0.3533	-0.5454	-0.1612
6 - 11	-0.1488	0.0433	0.0433	-0.1488	0.2354
6 - 1	0.0092	0.1293	0.1293	0.0092	0.2493
6 - 5	-0.0331	0.1590	0.1590	-0.0331	0.3511
6 - 3	-0.1178	0.1757	0.1757	-0.1178	0.4691
6 - 4	0.0359	0.1762	0.1762	0.0359	0.3165
6 - 7	-0.0121	0.1800	0.1800	-0.0121	0.3721
6 - 12	0.0188	0.1942	0.1942	0.0188	0.3695
6 - 10	-0.0938	0.1997	0.1997	-0.0938	0.4931
11 - 9	-0.6185	-0.3967	-0.3967	-0.6185	-0.1748
11 - 6	-0.2354	-0.0433	-0.0433	-0.2354	0.1488
11 - 1	-0.0775	0.0859	0.0859	-0.0775	0.2494
11 - 5	-0.1062	0.1157	0.1157	-0.1062	0.3375
11 - 3	-0.1814	0.1323	0.1323	-0.1814	0.4460
11 - 4	-0.0460	0.1328	0.1328	-0.0460	0.3117
11 - 7	-0.0852	0.1367	0.1367	-0.0852	0.3585
11 - 12	-0.0567	0.1508	0.1508	-0.0567	0.3583
11 - 10	-0.1574	0.1563	0.1563	-0.1574	0.4700
1 - 9	-0.6460	-0.4826	-0.4826	-0.6460	-0.3192
1 - 6	-0.2493	-0.1293	-0.1293	-0.2493	-0.0092
1 - 11	-0.2494	-0.0859	-0.0859	-0.2494	0.0775
1 - 5	-0.1337	0.0297	0.0297	-0.1337	0.1932
1 - 3	-0.2291	0.0464	0.0464	-0.2291	0.3219
1 - 4	-0.0505	0.0469	0.0469	-0.0505	0.1443
1 - 7	-0.1127	0.0507	0.0507	-0.1127	0.2142
1 - 12	-0.0785	0.0649	0.0649	-0.0785	0.2083
1 - 10	-0.2051	0.0704	0.0704	-0.2051	0.3459

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
12 - 10	-0.2982	0.3092	0.0055	-0.2982	0.3092
10 - 9	-0.8667	-0.2393	-0.5530	-0.8667	-0.2393
10 - 6	-0.4931	0.0938	-0.1997	-0.4931	0.0938
10 - 11	-0.4700	0.1574	-0.1563	-0.4700	0.1574
10 - 1	-0.3459	0.2051	-0.0704	-0.3459	0.2051
10 - 5	-0.3544	0.2730	-0.0407	-0.3544	0.2730
10 - 3	-0.4082	0.3602	-0.0240	-0.4082	0.3602
10 - 4	-0.3084	0.2614	-0.0235	-0.3084	0.2614
10 - 7	-0.3334	0.2940	-0.0197	-0.3334	0.2940
10 - 12	-0.3092	0.2982	-0.0055	-0.3092	0.2982

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
5 - 9	-0.7342	-0.5123	-0.5123	-0.2905	***
5 - 6	-0.3511	-0.1590	-0.1590	0.0331	***
5 - 11	-0.3375	-0.1157	-0.1157	0.1062	***
5 - 1	-0.1932	-0.0297	-0.0297	0.1337	***
5 - 3	-0.2970	0.0167	0.0167	0.3304	***
5 - 4	-0.1617	0.0172	0.0172	0.1960	***
5 - 7	-0.2008	0.0210	0.0210	0.2428	***
5 - 12	-0.1723	0.0352	0.0352	0.2427	***
5 - 10	-0.2730	0.0407	0.0407	0.3544	***
3 - 9	-0.8427	-0.5290	-0.5290	-0.2153	***
3 - 6	-0.4691	-0.1757	-0.1757	0.1178	***
3 - 11	-0.4460	-0.1323	-0.1323	0.1814	***
3 - 1	-0.3219	-0.0464	-0.0464	0.2291	***
3 - 5	-0.3304	-0.0167	-0.0167	0.2970	***
3 - 4	-0.2844	0.0005	0.0005	0.2854	***
3 - 7	-0.3094	0.0043	0.0043	0.3180	***
3 - 12	-0.2852	0.0185	0.0185	0.3222	***
3 - 10	-0.3602	0.0240	0.0240	0.4082	***
4 - 9	-0.7083	-0.5295	-0.5295	-0.3507	***
4 - 6	-0.3165	-0.1762	-0.1762	-0.0359	***
4 - 11	-0.3117	-0.1328	-0.1328	0.0460	***
4 - 1	-0.1443	-0.0469	-0.0469	0.0505	***
4 - 5	-0.1960	-0.0172	-0.0172	0.1617	***
4 - 3	-0.2854	-0.0005	-0.0005	0.2844	***
4 - 7	-0.1750	0.0038	0.0038	0.1827	***
4 - 12	-0.1427	0.0180	0.0180	0.1787	***
4 - 10	-0.2614	0.0235	0.0235	0.3084	***
7 - 9	-0.7552	-0.5333	-0.5333	-0.3115	***
7 - 6	-0.3721	-0.1800	-0.1800	0.0121	***
7 - 11	-0.3585	-0.1367	-0.1367	0.0852	***
7 - 1	-0.2142	-0.0507	-0.0507	0.1127	***
7 - 5	-0.2428	-0.0210	-0.0210	0.2008	***
7 - 3	-0.3180	-0.0043	-0.0043	0.3094	***
7 - 4	-0.1827	-0.0038	-0.0038	0.1750	***
7 - 12	-0.1933	0.0142	0.0142	0.2217	***
7 - 10	-0.2940	0.0197	0.0197	0.3334	***
12 - 9	-0.7550	-0.5475	-0.5475	-0.3400	***
12 - 6	-0.3695	-0.1942	-0.1942	-0.0188	***
12 - 11	-0.3583	-0.1508	-0.1508	0.0567	***
12 - 1	-0.2083	-0.0649	-0.0649	0.0785	***
12 - 5	-0.2427	-0.0352	-0.0352	0.1723	***
12 - 3	-0.3222	-0.0185	-0.0185	0.2852	***
12 - 4	-0.1787	-0.0180	-0.0180	0.1427	***
12 - 7	-0.2217	-0.0142	-0.0142	0.1933	***

18. CATION EXCHANGE CAPACITY CEC

units: cmol+/kg

mandatory for samples A1, B1 and B2
only demanded for B1 and B2
reported by 18 laboratories

TABLE I : CEC - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2	A1 B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5	1 not reported		N/R
6	B1 B2	2	R
7	A1 B1 B2	3	N/R
8	B1 B2	3	N/R
9	A1 B1 B2	3	R
10	B1 B2	3	R
11			
12	B1 B2	7	N
13	A1 A2 B1 B2	3	N
14	A1 B1 B2	5	N/R
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	1 not reported	N
19	B1 B2	3	N=R
20			
21	A1 B1 B2	3	N/R
22			

TABLE II : CEC - Summary method descriptions

method	description
1	reference
2	1N Na2Ac at pH 7, replace with NH4+Ac at pH 7
3	1M NH4NO3, sum exch.cations
4	NH4Cl, sum exch.cations
5	AFNOR X31-130
6	back-exchange with 0.2 mol/l HCl
7	0.01M AgTu
8	sum(Ca,Mg,Na,K,H)
9	0.1M BaCl2 at pH 8.1, 1N NH4Cl at pH 8
10	0.1M BaCl2, 1M NH4Cl
11	BaCl2 at pH 8.1, exchanged by MgSO4
12	sum exchangeable cations and soil unsaturation
13	unspecified

TABLE III a : CEC - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
2	2	3	20.930	21.780	22.840	0.972	4.464
3	3	5	34.910	35.218	35.470	0.200	0.568
7	5	3	18.910	19.013	19.130	0.111	0.582
9	6	3	20.100	20.100	20.100	0.000	0.000
13	7	3	22.805	23.231	23.797	0.510	2.197
14	8	5	20.510	20.742	20.940	0.161	0.775
21	11	3	6.700	9.467	12.200	2.750	29.051

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	3	5	68.270	69.119	69.750	0.680	0.984
13	7	3	45.224	47.310	50.107	2.518	5.322

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	12.200	12.620	12.830	0.364	2.882
3	3	5	8.730	8.803	8.870	0.050	0.568
4	4	4	8.985	9.149	9.522	0.250	2.734
5	13	1		8.210			
6							
7	5	3	7.760	7.913	8.220	0.266	3.356
8	13	3	4.920	5.260	5.860	0.507	9.605
9	6	3	8.300	8.333	8.400	0.058	0.693
10							
11							
12	12	7	11.940	12.350	12.690	0.285	2.308
13	7	3	8.933	8.956	8.956	0.034	0.382
14	8	5	6.822	6.917	6.979	0.060	0.873
15							
16	4	3	9.080	9.163	9.220	0.074	0.804
17	13	3	9.880	9.923	9.950	0.038	0.382
18	10	1		15.700			
19							
20							
21	11	3	3.700	5.533	7.200	1.756	31.734
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	22.840	24.740	26.640	1.900	7.680
3	3	5	5.640	5.781	5.870	0.090	1.557
4	4	4	4.698	4.826	4.981	0.129	2.669
5	13	1		5.190			
6							
7	5	3	3.840	3.967	4.100	0.130	3.281
8	13	3	3.590	3.757	3.890	0.153	4.066
9	6	3	7.100	7.200	7.400	0.173	2.406
10							
11							
12	12	6	24.410	25.443	27.010	1.068	4.199
13	7	3	5.577	5.897	6.456	0.486	8.238
14	8	5	5.423	5.607	6.147	0.306	5.462
15							
16	4	3	3.850	4.420	4.730	0.494	11.183
17	13	3	6.540	6.587	6.650	0.057	0.863
18	10	1	4.800	4.800	4.800		
19							
20							
21	11	3	12.300	14.933	18.500	3.204	21.453
22							

TABLE III b : CEC - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
9	3	20.30	20.33	20.40	0.06	0.28
14	5	20.52	20.75	20.95	0.16	0.77
19	3		265.40			
21	3	27.90	28.57	29.00	0.59	2.05

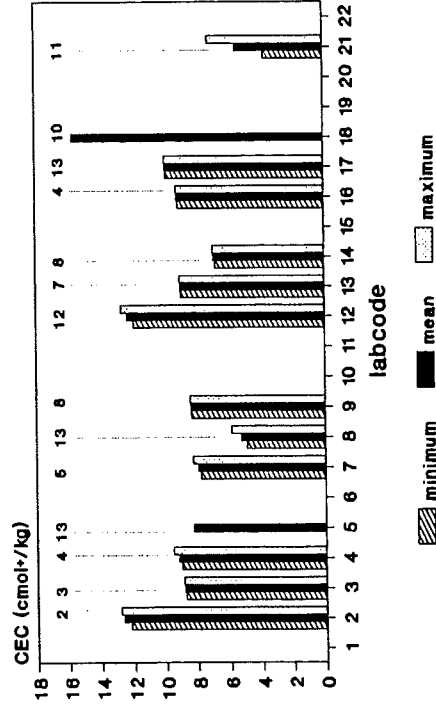
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	8.30	8.45	8.81	0.24	2.87
5	1	9.42	9.42	9.42		
6	2	6.29	6.30	6.30	0.01	0.11
7	3	7.05	7.12	7.23	0.10	1.39
8	3	4.40	4.55	4.80	0.22	4.74
9	3	8.06	8.12	8.20	0.07	0.89
10	3	7.41	7.43	7.45	0.02	0.27
11						
12						
13						
14	5	6.75	6.79	6.91	0.07	1.08
15	3	7.18	7.32	7.48	0.15	2.06
16	3	6.94	6.96	6.99	0.03	0.38
17						
18						
19	3		76.50			
20						
21	3	9.56	9.89	10.20	0.32	3.24
22						

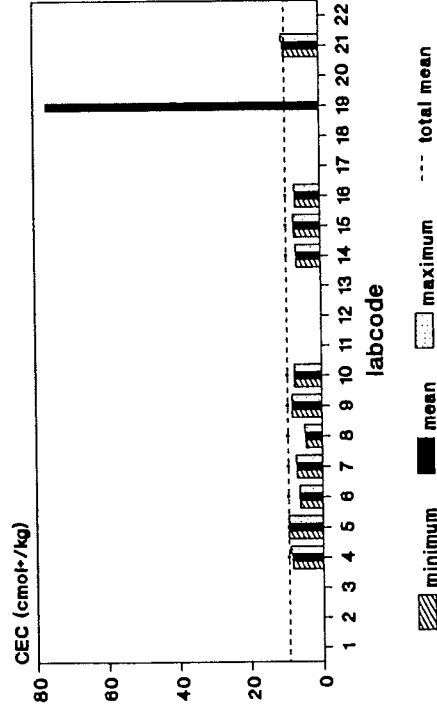
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	6.22	6.30	6.38	0.08	1.21
5	1		4.99			
6	1	3.47	3.60	3.72	0.18	4.92
7	3	4.75	4.87	4.93	0.10	2.08
8	3	3.22	3.42	3.56	0.18	5.16
9	3	6.36	6.57	6.68	0.18	2.71
10	3	5.71	5.77	5.83	0.06	1.04
11						
12						
13						
14	5	4.94	5.12	5.64	0.29	5.75
15	3	5.27	5.44	5.55	0.15	2.75
16	3	5.21	5.24	5.27	0.03	0.58
17						
18						
19	3		56.80			
20						
21	3	7.64	8.10	8.68	0.53	6.56
22						

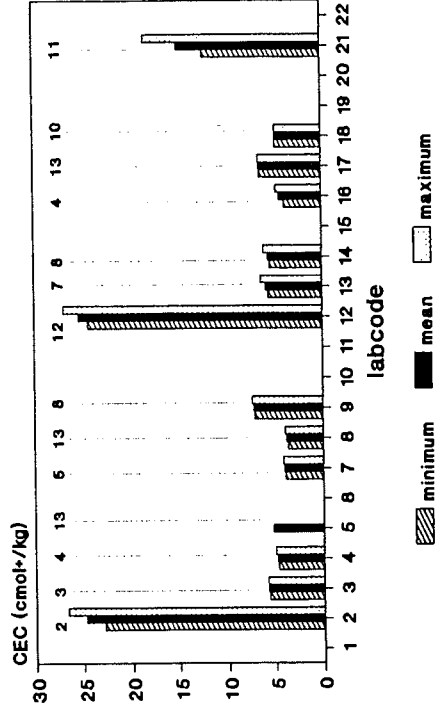
CEC sample B1
national methods



CEC sample B1
reference method



CEC sample B2
national methods



CEC sample B2
reference method

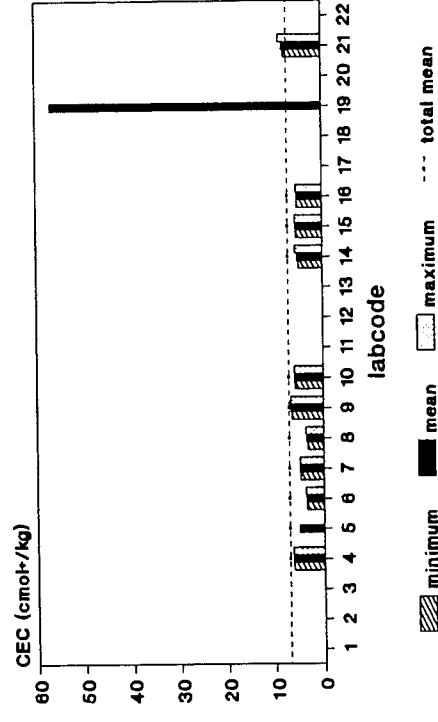


TABLE V : CEC (cmol+/kg) Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 11 2 3 5 6 7 8

Number of observations in by group = 33

Dependent Variable: CEC

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	4651.19993088	0.31	0.9440
Error	25	54121.82839100		

Corrected Total

58773.02832188

CEC Mean

C.V.

R-Square

Type I SS

F Value

Pr > F

0.079138 164.1203 28.35006061

Source	DF	Type III SS	F Value	Pr > F
METH	7	4651.19993088	0.31	0.9440
Source	DF	Type III SS	F Value	Pr > F
METH	7	4651.19993088	0.31	0.9440

TABLE IV : CEC - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	12	42.988	70.132	0.358	0.000
A1	2	3	21.780	0.972	0.965	0.641
A1	3	1	35.218			
A1	5	3	19.013	0.111	0.989	0.800
A1	6	3	20.100	0.000		
A1	7	3	23.231	0.510	0.944	0.544
A1	8	5	20.742	0.161	0.987	0.956
A1	11	3	9.467	2.750	1.000	0.978
A2	3	1	69.119			
A2	7	3	47.310	2.518	0.940	0.529
B1	1	34	9.425	11.927	0.265	0.000
B1	2	3	12.620	0.364	0.750	0.000
B1	3	1	8.803			
B1	4	7	9.155	0.182	0.842	0.106
B1	5	3	7.913	0.266	0.750	0.000
B1	6	3	8.333	0.058	0.750	0.000
B1	7	3	8.956	0.034	0.822	0.168
B1	8	5	6.917	0.060	0.932	0.619
B1	10	1	15.700			
B1	11	3	5.533	1.756	0.993	0.843
B1	12	7	12.350	0.285	0.914	0.441
B1	13	7	7.689	2.351	0.803	0.044
B2	1	34	6.998	8.886	0.288	0.000
B2	2	3	24.740	1.900	1.000	0.992
B2	3	1	5.781			
B2	4	7	4.652	0.370	0.730	0.008
B2	5	3	3.967	0.130	0.998	0.915
B2	6	3	7.200	0.173	0.750	0.000
B2	7	3	5.897	0.486	0.818	0.159
B2	8	5	5.607	0.306	0.688	0.008
B2	10	1	4.800			
B2	11	3	14.933	3.204	0.936	0.513
B2	12	6	25.443	1.068	0.874	0.237
B2	13	7	5.174	1.418	0.803	0.044

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.

To calculate the total number of replicates for the method groups, SAS counts the number of reported results.

So, the "repl" values in this table for methods 3 and 1 are different from the real number of replicates.

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
6 - 1	-122.00	-22.89	76.23
6 - 3	-192.42	-15.12	162.18
6 - 7	-128.50	-3.13	122.24
6 - 2	-127.05	-1.68	123.69
6 - 8	-112.78	-0.64	111.49
6 - 5	-124.29	1.09	126.46
6 - 11	-114.74	10.63	136.01
5 - 1	-123.09	-23.97	75.14
5 - 3	-193.51	-16.20	161.10
5 - 7	-129.59	-4.22	121.15
5 - 2	-128.14	-2.77	122.61
5 - 8	-113.86	-1.73	110.41
5 - 6	-126.46	-1.09	124.29
5 - 11	-115.83	9.55	134.92
11 - 1	-132.64	-33.52	65.59
11 - 3	-203.05	-25.75	151.55
11 - 7	-139.14	-13.76	111.61
11 - 2	-137.69	-12.31	113.06
11 - 8	-123.41	-11.28	100.86
11 - 6	-136.01	-10.63	114.74
11 - 5	-134.92	-9.55	115.83

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CEC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 25 MSE= 2164.873
Critical Value of Studentized Range= 4.667

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
1 - 3	-152.05	7.77	167.59
1 - 7	-79.36	19.76	118.87
1 - 2	-77.91	21.21	120.32
1 - 8	-59.49	22.25	103.98
1 - 6	-76.23	22.89	122.00
1 - 5	-75.14	23.97	123.09
1 - 11	-65.59	33.52	132.64
3 - 1	-167.59	-7.77	152.05
3 - 7	-165.32	11.99	189.29
3 - 2	-163.86	13.44	190.74
3 - 8	-153.73	14.48	182.68
3 - 6	-162.18	15.12	192.42
3 - 5	-161.10	16.20	193.51
3 - 11	-151.55	25.75	203.05
7 - 1	-118.87	-19.76	79.36
7 - 3	-189.29	-11.99	165.32
7 - 2	-123.92	1.45	126.82
7 - 8	-109.65	2.49	114.63
7 - 6	-122.24	3.13	128.50
7 - 5	-121.15	4.22	129.59
7 - 11	-111.61	13.76	139.14
2 - 1	-120.32	-21.21	77.91
2 - 3	-190.74	-13.44	163.86
2 - 7	-126.82	-1.45	123.92
2 - 8	-111.10	1.04	113.17
2 - 6	-123.69	1.68	127.05
2 - 5	-122.61	2.77	128.14
2 - 11	-113.06	12.31	137.69
8 - 1	-103.98	-22.25	59.49
8 - 3	-182.68	-14.48	153.73
8 - 7	-114.63	-2.49	109.65
8 - 2	-113.17	-1.04	111.10
8 - 6	-111.49	0.64	112.78
8 - 5	-110.41	1.73	113.86
8 - 11	-100.86	11.28	123.41

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	12	1 10 11 12 13 2 3 4 5 6 7 8

Number of observations in by group = 77

Dependent Variable: CEC

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	238.08484304	0.30	0.9842
Error	65	4734.46961304		
Corrected Total	76	4972.55445608		

R-Square C.V. CEC Mean
0.047880 92.04240 9.27237662

Source	DF	Type I SS	F Value	Pr > F
METH	11	238.08484304	0.30	0.9842
Source	DF	Type III SS	F Value	Pr > F
METH	11	238.08484304	0.30	0.9842

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	2	3 7

Number of observations in by group = 4

Dependent Variable: CEC

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	356.71345633	56.27	0.0173
Error	2	12.67870467		
Corrected Total	3	369.39216100		

R-Square C.V. CEC Mean
0.965677 4.771963 52.76250000

Source	DF	Type I SS	F Value	Pr > F
METH	1	356.71345633	56.27	0.0173
Source	DF	Type III SS	F Value	Pr > F
METH	1	356.71345633	56.27	0.0173

Tukey's Studentized Range (HSD) Test for variable: CEC

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CEC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 65 MSE= 72.83799
Critical Value of Studentized Range= 4.794

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison		Simultaneous Lower Confidence Limit			Difference Between Means			Simultaneous Upper Confidence Limit		
METH Comparison		Simultaneous Lower Confidence Limit			Difference Between Means			Simultaneous Upper Confidence Limit		
10	- 2	-30.327	3.080	36.487	3.080	3.350	36.487	-30.327	3.080	36.487
10	- 12	-27.579	3.350	34.279	3.350	3.350	34.279	-27.579	3.350	34.279
10	- 1	-23.079	6.275	35.628	6.275	6.275	35.628	-23.079	6.275	35.628
10	- 4	-24.384	6.545	37.473	6.545	6.545	37.473	-24.384	6.545	37.473
10	- 7	-26.663	6.744	40.151	6.744	6.744	40.151	-26.663	6.744	40.151
10	- 3	-34.018	6.897	47.812	6.897	6.897	47.812	-34.018	6.897	47.812
10	- 6	-26.040	7.367	40.774	7.367	7.367	40.774	-26.040	7.367	40.774
10	- 5	-25.620	7.787	41.194	7.787	7.787	41.194	-25.620	7.787	41.194
10	- 13	-22.917	8.011	38.940	8.011	8.011	38.940	-22.917	8.011	38.940
10	- 8	-22.909	8.783	40.476	8.783	8.783	40.476	-22.909	8.783	40.476
10	- 11	-23.240	10.167	43.574	10.167	10.167	43.574	-23.240	10.167	43.574
2	- 10	-36.487	-3.080	30.327	-3.080	0.270	30.327	-36.487	-3.080	30.327
2	- 12	-19.694	0.270	20.234	0.270	0.470	20.234	-19.694	0.470	20.234
2	- 1	-14.230	3.195	20.619	3.195	0.622	20.619	-14.230	0.622	20.619
2	- 4	-16.500	3.465	23.429	3.465	1.092	23.429	-16.500	1.092	23.429
2	- 7	-19.958	3.664	27.287	3.664	1.512	27.287	-19.958	1.512	27.287
2	- 3	-29.590	3.817	37.224	3.817	1.737	37.224	-29.590	1.737	37.224
2	- 6	-19.336	4.287	27.909	4.287	2.509	27.909	-19.336	2.509	27.909
2	- 5	-18.916	4.707	28.329	4.707	3.892	28.329	-18.916	3.892	28.329
2	- 13	-15.033	4.931	24.896	4.931	3.422	24.896	-15.033	3.422	24.896
2	- 8	-15.425	5.703	26.832	5.703	3.622	26.832	-15.425	3.622	26.832
2	- 11	-16.536	7.087	30.709	7.087	3.622	30.709	-16.536	3.622	30.709
12	- 10	-34.279	-3.350	27.579	-3.350	-6.897	27.579	-34.279	-6.897	27.579
12	- 2	-20.234	-0.270	19.694	-0.270	-3.817	19.694	-20.234	-3.817	19.694
12	- 1	-9.083	2.925	14.933	2.925	-3.547	14.933	-9.083	-3.547	14.933
12	- 4	-12.270	3.195	18.659	3.195	-3.547	18.659	-12.270	-3.547	18.659
12	- 7	-16.570	3.394	23.359	3.394	-0.622	23.359	-16.570	-0.622	23.359
12	- 3	-27.382	3.547	34.476	3.547	-0.352	34.476	-27.382	-0.352	34.476
12	- 6	-15.948	4.017	23.981	4.017	-0.153	23.981	-15.948	-0.153	23.981
12	- 5	-15.528	4.437	24.401	4.437	0.470	24.401	-15.528	0.470	24.401
12	- 13	-10.803	4.661	20.126	4.661	0.890	20.126	-10.803	0.890	20.126
12	- 8	-11.507	5.433	22.374	5.433	1.114	22.374	-11.507	1.114	22.374
12	- 11	-13.148	6.817	26.781	6.817	1.886	26.781	-13.148	1.886	26.781
1	- 10	-35.628	-6.275	23.079	-6.275	-7.367	23.079	-35.628	-7.367	23.079
1	- 2	-20.619	-3.195	14.230	-3.195	-4.287	14.230	-20.619	-4.287	14.230
1	- 12	-14.933	-2.925	9.083	-2.925	-4.017	9.083	-14.933	-4.017	9.083

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
11 - 1	-21.317	13.533	-3.892	-21.317	13.533
11 - 4	-23.586	16.342	-3.622	-23.586	16.342
11 - 7	-27.045	20.200	-3.422	-27.045	20.200
11 - 3	-36.677	30.137	-3.270	-36.677	30.137
11 - 6	-26.422	20.822	-2.800	-26.422	20.822
11 - 5	-26.002	21.242	-2.380	-26.002	21.242
11 - 13	-22.120	17.809	-2.155	-22.120	17.809
11 - 8	-22.512	19.745	-1.383	-22.512	19.745

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
6 - 1	-18.517	16.333	-1.092	-18.517	16.333
6 - 4	-20.786	19.142	-0.822	-20.786	19.142
6 - 7	-24.245	23.000	-0.622	-24.245	23.000
6 - 3	-33.877	32.937	-0.470	-33.877	32.937
6 - 5	-23.202	24.042	0.420	-23.202	24.042
6 - 13	-19.320	20.609	0.645	-19.320	20.609
6 - 8	-19.712	22.545	1.417	-19.712	22.545
6 - 11	-20.822	26.422	2.800	-20.822	26.422
5 - 10	-41.194	25.620	-7.787	-41.194	25.620
5 - 2	-28.329	18.916	-4.707	-28.329	18.916
5 - 12	-24.401	15.528	-4.437	-24.401	15.528
5 - 1	-18.937	15.913	-1.512	-18.937	15.913
5 - 4	-21.206	18.722	-1.242	-21.206	18.722
5 - 7	-24.665	22.580	-1.042	-24.665	22.580
5 - 3	-34.297	32.517	-0.890	-34.297	32.517
5 - 6	-24.042	23.202	-0.420	-24.042	23.202
5 - 13	-19.740	20.189	0.225	-19.740	20.189
5 - 8	-20.132	22.125	0.997	-20.132	22.125
5 - 11	-21.242	26.002	2.380	-21.242	26.002
13 - 10	-38.940	22.917	-8.011	-38.940	22.917
13 - 2	-24.896	15.033	-4.931	-24.896	15.033
13 - 12	-20.126	10.803	-4.661	-20.126	10.803
13 - 1	-13.745	10.271	-1.737	-13.745	10.271
13 - 4	-16.931	13.998	-1.467	-16.931	13.998
13 - 7	-21.232	18.697	-1.267	-21.232	18.697
13 - 3	-32.043	29.814	-1.114	-32.043	29.814
13 - 6	-20.609	19.320	-0.645	-20.609	19.320
13 - 5	-20.189	19.740	-0.225	-20.189	19.740
13 - 8	-16.168	17.712	0.772	-16.168	17.712
13 - 11	-17.809	22.120	2.155	-17.809	22.120
8 - 10	-40.476	22.909	-8.783	-40.476	22.909
8 - 2	-26.832	15.425	-5.703	-26.832	15.425
8 - 12	-22.374	11.507	-5.433	-22.374	11.507
8 - 1	-16.366	11.348	-2.509	-16.366	11.348
8 - 4	-19.179	14.702	-2.239	-19.179	14.702
8 - 7	-23.167	19.089	-2.039	-23.167	19.089
8 - 3	-33.579	29.806	-1.886	-33.579	29.806
8 - 6	-22.545	19.712	-1.417	-22.545	19.712
8 - 5	-22.125	20.132	-0.997	-22.125	20.132
8 - 13	-17.712	16.168	-0.772	-17.712	16.168
8 - 11	-19.745	22.512	1.383	-19.745	22.512
11 - 10	-43.574	23.240	-10.167	-43.574	23.240
11 - 2	-30.709	16.536	-7.087	-30.709	16.536
11 - 12	-26.781	13.148	-6.817	-26.781	13.148

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	12	1 10 11 12 13 2 3 4 5 6 7 8

Number of observations in by group = 76

Dependent Variable: CEC

Source	DF	Sum of Squares	F Value	Pr > F
Model	11	3039.06901643	6.67	0.0001
Error	64	2652.89977077		
Corrected Total	75	5691.96878720		

R-Square	C.V.	CEC Mean
0.533922	73.22664	8.79227632

Source	DF	Type I SS	F Value	Pr > F
METH	11	3039.06901643	6.67	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	11	3039.06901643	6.67	0.0001

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CEC

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 41.45156
Critical Value of Studentized Range= 4.797

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit			
12 - 2	-14.738	0.703	16.145		
12 - 11	-4.931	10.510	25.951		
12 - 6	2.802	18.243	33.685	***	
12 - 1	8.775	18.445	28.115	***	
12 - 7	4.105	19.546	34.988	***	
12 - 3	-3.925	19.662	43.250	***	
12 - 8	6.613	19.836	33.059	***	
12 - 13	8.120	20.269	32.418	***	
12 - 10	-2.944	20.643	44.231	***	
12 - 4	8.642	20.791	32.941	***	
12 - 5	6.035	21.477	36.918	***	
2 - 12	-16.145	-0.703	14.738		
2 - 11	-8.024	9.807	27.637		
2 - 6	-0.290	17.540	35.370	***	
2 - 1	4.589	17.742	30.894	***	
2 - 7	1.013	18.843	36.673	***	
2 - 3	-6.257	18.959	44.175	***	
2 - 8	3.185	19.133	35.080	***	
2 - 13	4.496	19.566	34.635	***	
2 - 10	-5.276	19.940	45.156	***	
2 - 4	5.019	20.088	35.157	***	
2 - 5	2.943	20.773	38.604	***	
11 - 12	-25.951	-10.510	4.931		
11 - 2	-27.637	-9.807	8.024		
11 - 6	-10.097	7.733	25.564		
11 - 1	-5.217	7.935	21.087		
11 - 7	-8.794	9.036	26.867		
11 - 3	-16.063	9.152	34.368		
11 - 8	-6.622	9.326	25.274		
11 - 13	-5.310	9.759	24.828		
11 - 10	-15.082	10.133	35.349		
11 - 4	-4.788	10.281	25.351		
11 - 5	-6.864	10.967	28.797		
6 - 12	-33.685	-18.243	-2.802	***	
6 - 2	-35.370	-17.540	0.290		
6 - 11	-25.564	-7.733	10.097		

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous			Difference Between Means	Simultaneous		
	Lower Confidence Limit	Upper Confidence Limit			Lower Confidence Limit	Upper Confidence Limit	
6 - 1	-12.951	13.354	0.202	-18.445	-8.775	***	***
6 - 7	-16.527	19.133	1.303	-17.742	-4.589	***	***
6 - 3	-23.797	26.635	1.419	-7.935	5.217		
6 - 8	-14.355	17.540	1.593	-0.202	12.951		
6 - 13	-13.044	17.095	2.026	1.101	14.254		
6 - 10	-22.816	27.616	2.400	1.217	23.374		
6 - 4	-12.521	17.617	2.548	1.391	11.850		
6 - 5	-14.597	21.064	3.233	1.824	10.888		
1 - 12	-28.115	-8.775	-18.445	1.391	11.850		
1 - 2	-30.894	-4.589	-17.742	1.824	10.888		
1 - 11	-21.087	5.217	-7.935	2.198	24.355		
1 - 6	-13.354	12.951	-0.202	2.346	11.410		
1 - 7	-12.051	14.254	1.101	3.032	16.184		
1 - 3	-20.939	23.374	1.217				
1 - 8	-9.069	11.850	1.391				
1 - 13	-7.240	10.888	1.824				
1 - 10	-19.958	24.355	2.198				
1 - 4	-6.717	11.410	2.346				
1 - 5	-10.121	16.184	3.032				
7 - 12	-34.988	-4.105	-19.546			***	***
7 - 2	-36.673	-1.013	-18.843			***	***
7 - 11	-26.867	8.794	-9.036				
7 - 6	-19.133	16.527	-1.303				
7 - 1	-14.254	12.051	-1.101				
7 - 3	-25.100	25.332	0.116				
7 - 8	-15.658	16.237	0.290				
7 - 13	-14.347	15.792	0.723				
7 - 10	-24.119	26.313	1.097				
7 - 4	-13.824	16.314	1.245				
7 - 5	-15.900	19.761	1.930				
3 - 12	-43.250	3.925	-19.662				
3 - 2	-44.175	6.257	-18.959				
3 - 11	-34.368	16.063	-9.152				
3 - 6	-26.635	23.797	-1.419				
3 - 1	-23.374	20.939	-1.217				
3 - 7	-25.332	25.100	-0.116				
3 - 8	-23.748	24.095	0.174				
3 - 13	-22.739	23.952	0.607				
3 - 10	-29.902	31.864	0.981				
3 - 4	-22.216	24.474	1.129				
3 - 5	-23.401	27.030	1.814				
8 - 12	-33.059	-6.613	-19.836			***	***
8 - 2	-35.080	-3.185	-19.133			***	***
8 - 11	-25.274	6.622	-9.326				

General Linear Models Procedure

METH Comparison	Simultaneous			Difference Between Means	Simultaneous		
	Lower Confidence Limit	Upper Confidence Limit			Lower Confidence Limit	Upper Confidence Limit	
8 - 6	-17.540	14.355	-1.593	-20.269	-8.120	***	***
8 - 1	-11.850	9.069	-1.391	-19.566	-4.496	***	***
8 - 7	-16.237	15.658	-0.290	-9.759	5.310		
8 - 3	-24.095	23.748	-0.174	-2.026	13.044		
8 - 13	-12.354	0.433	0.433	-1.824	7.240		
8 - 10	-23.114	0.807	0.807	-0.723	14.347		
8 - 4	-11.831	13.742	0.956	-0.607	22.739		
8 - 5	-14.307	17.589	1.641	-0.433	12.354		
13 - 12	-32.418	-8.120	-20.269	-0.433	12.354		
13 - 2	-34.635	-4.496	-19.566	0.374	23.720		
13 - 11	-24.828	5.310	-9.759	0.522	12.195		
13 - 6	-17.095	13.044	-2.026	1.208	16.277		
13 - 1	-10.888	7.240	-1.824				
13 - 7	-15.792	14.347	-0.723				
13 - 3	-23.952	22.739	-0.607				
13 - 8	-13.220	12.354	-0.433				
13 - 10	-22.971	0.374	0.374				
13 - 4	-11.150	12.195	0.522				
13 - 5	-13.862	16.277	1.208				
10 - 12	-44.231	2.944	-20.643				
10 - 2	-45.156	5.276	-19.940				
10 - 11	-35.349	15.082	-10.133				
10 - 6	-27.616	22.816	-2.400				
10 - 1	-24.355	19.958	-2.198				
10 - 7	-26.313	24.119	-1.097				
10 - 3	-31.864	29.902	-0.981				
10 - 8	-24.729	23.114	-0.807				
10 - 13	-23.720	22.971	-0.374				
10 - 4	-23.197	23.493	0.148				
10 - 5	-24.382	26.049	0.833				
4 - 12	-32.941	-8.642	-20.791			***	***
4 - 2	-35.157	-5.019	-20.088			***	***
4 - 11	-25.351	4.788	-10.281				
4 - 6	-17.617	12.521	-2.548				
4 - 1	-11.410	6.717	-2.346				
4 - 7	-16.314	13.824	-1.245				
4 - 3	-24.474	22.216	-1.129				
4 - 8	-13.742	11.831	-0.956				
4 - 13	-12.195	11.150	-0.522				
4 - 10	-23.493	23.197	-0.148				
4 - 5	-14.384	15.755	0.685				
5 - 12	-36.918	-6.035	-21.477			***	***
5 - 2	-38.604	-2.943	-20.773			***	***
5 - 11	-28.797	6.864	-10.967				

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	
5 - 6	-21.064	14.597	-3.233	14.597	
5 - 1	-16.184	10.121	-3.032	10.121	
5 - 7	-19.761	15.900	-1.930	15.900	
5 - 3	-27.030	23.401	-1.814	23.401	
5 - 8	-17.589	14.307	-1.641	14.307	
5 - 13	-16.277	13.862	-1.208	13.862	
5 - 10	-26.049	24.382	-0.833	24.382	
5 - 4	-15.755	14.384	-0.685	14.384	

19. BASE SATURATION

units: %

mandatory for samples A1, B1 and B2
only demanded for B1 and B2
reported by 20 laboratories

TABLE I : Base saturation - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1			
2	B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	B1 B2	4	N/R
5	B1 B2	1 not reported	N/R
6	B1 B2	2	R
7	B1 B2	3	N/R
8	B1 B2	3	N/R
9	A1 B1 B2	3	R
10	B1 B2	3	R
11			
12	B1 B2	7	N
13	A1 A2 B1 B2	3	N
14	B1 B2	5	N
15	B1 B2	3	R
16	B1 B2	3	N/R
17	B1 B2	3	N
18	B1 B2	1 not reported	N
19	A1 B1 B2	3	N=R
20	A1 B1 B2	1 not reported	R
21	A1 B1 B2	3	N/R
22			

TABLE II : Base saturation - Summary method descriptions

method	description
1	reference
2	1N Na2Ac at pH 7, replace with NH4+Ac at pH 7
3	1M NH4NO3, sum exch.cations
4	NH4Cl, sum exch.cations
5	AFNOR X31-130
6	back-exchange with 0.2 mol/l HCl
7	0.01M AgTU
8	sum(Ca,Mg,Na,K,H)
9	0.1M BaCl2 at pH 8.1, 1N NH4Cl at pH 8
10	0.1M BaCl2, 1M NH4Cl
11	BaCl2 at pH 8.1, exchanged by MgSO4
12	sum exchangeable cations and soil unsaturation
13	unspecified

TABLE IIIa: Base saturation - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	3	5	99.920	99.930	99.930	0.000	0.000
13	7	3	100.000	100.000	100.000	0.000	0.000
14	8	5	100.000	100.000	100.000	0.000	0.000
20	13	1	99.910	99.910			
21	11	3	100.000	100.000	100.000	0.000	0.000

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	3	5	90.400	90.720	90.900	0.200	0.220
13	7	3	76.420	60.887	84.990	4.297	5.312

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	24.000	26.033	28.300	2.159	8.295
2	3	5	33.380	33.490	33.670	0.110	0.328
3	4	4	27.520	27.897	28.300	0.337	1.209
5	14	1	31.300	31.300			
6							
7	5	3	35.700	37.333	38.400	1.436	3.848
8	14	3	41.300	46.237	49.790	4.411	9.540
9							
10							
11							
12	12	7	24.800	29.071	32.300	2.642	9.089
13	7	3	27.260	28.040	28.470	0.677	2.413
14	8	5	40.200	40.640	41.200	0.378	0.930
15							
16	4	3	31.200	31.900	32.400	0.624	1.958
17	14	3	47.200	47.433	47.700	0.252	0.531
18	10	1	54.774	54.774			
19							
20	13	1	45.000	45.000			
21	11	3	86.250	86.757	87.320	0.537	0.619
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2	2	3	7.500	8.100	9.100	0.872	10.783
3	3	5	47.860	48.490	49.400	0.640	1.320
4	4	4	40.930	43.258	44.710	1.746	4.036
5	14	1		47.000			
6							
7	5	3	50.400	50.800	51.500	0.608	1.197
8	14	3	54.100	56.323	58.320	2.119	3.762
9							
10							
11							
12	12	7	11.100	12.457	13.800	0.995	7.985
13	7	3	44.250	44.660	45.240	0.516	1.156
14	8	5	46.700	47.340	48.200	0.586	1.237
15							
16	4	3	35.100	36.500	38.800	2.007	5.500
17	14	3	67.900	68.100	68.400	0.265	0.389
18	10	1		66.813			
19							
20	13	1		18.780			
21	11	3	78.190	80.193	83.250	2.689	3.354
22							

TABLE IIIb : Base saturation - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
9	3	100.00	100.00	100.00	0.00	0.00
14	2	100.00	100.00	100.00	0.00	0.00
19	3	100.00	100.00	100.00	0.00	0.00
21	3	99.58	99.73	99.90	0.16	0.16

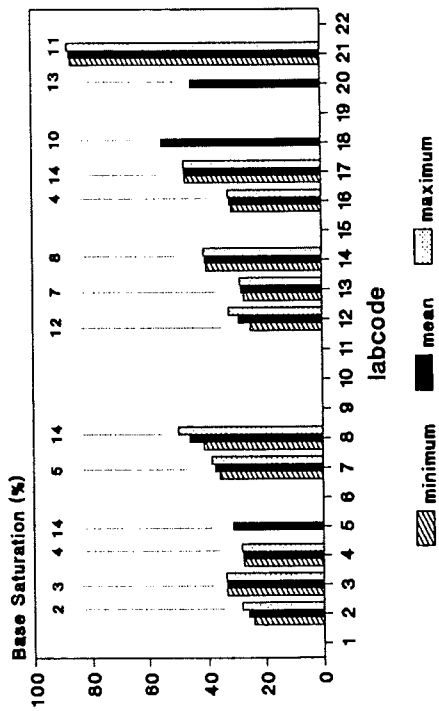
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	27.41	28.36	29.18	0.74	2.61
5	1		32.30			
6	2	41.00	41.70	42.40	0.99	2.37
7	3	41.00	41.33	41.90	0.49	1.19
8	3	47.04	50.34	53.36	3.17	6.30
9	3	28.40	28.97	29.40	0.51	1.77
10	3	42.10	42.27	42.50	0.21	0.49
11						
12						
13						
14	5	40.80	41.36	42.00	0.46	1.10
15	3	38.61	39.77	41.10	1.25	3.15
16	3	39.70	40.13	40.60	0.45	1.12
17						
18						
19	3		45.90			
20						
21	3	25.10	27.40	31.08	3.22	11.74
22						

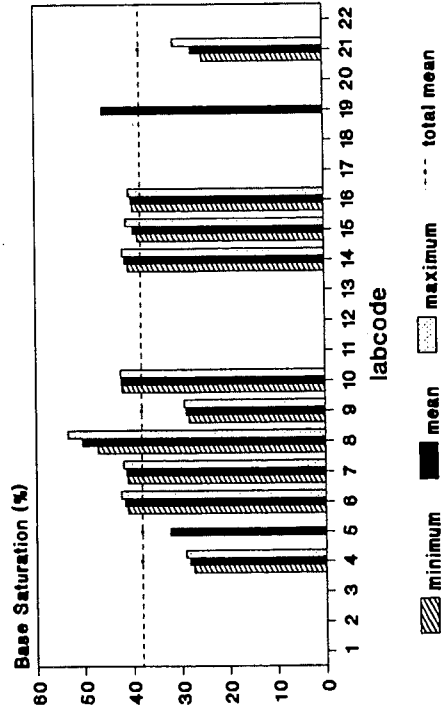
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	4	38.95	40.74	42.28	1.72	4.22
5	1		62.10			
6	2	58.90	59.40	59.90	0.71	1.19
7	3	50.80	51.47	52.10	0.65	1.26
8	3	53.65	55.89	58.69	2.57	4.59
9	3	37.40	38.07	38.50	0.59	1.54
10	3	50.20	51.07	51.80	0.81	1.58
11						
12						
13						
14	5	51.10	51.86	52.50	0.64	1.22
15	3	50.45	51.88	52.94	1.29	2.48
16	3	48.20	48.93	50.00	0.95	1.93
17						
18						
19	3		53.20			
20						
21	3	29.10	33.13	35.21	3.49	10.54
22						

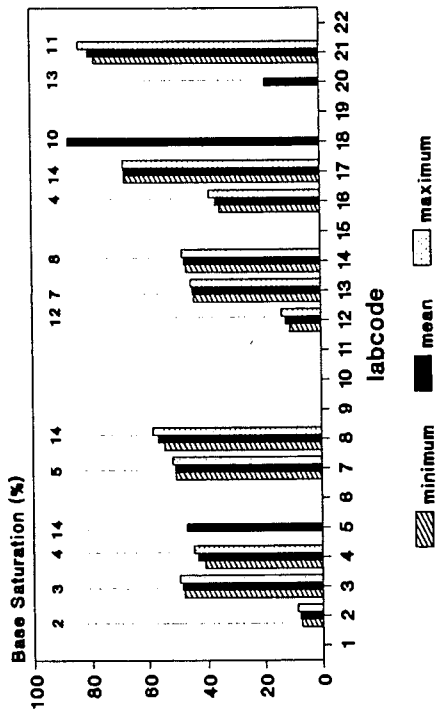
Base Saturation sample B1
national methods



Base Saturation sample B1
reference method



Base Saturation sample B2
national methods



Base Saturation sample B2
reference method

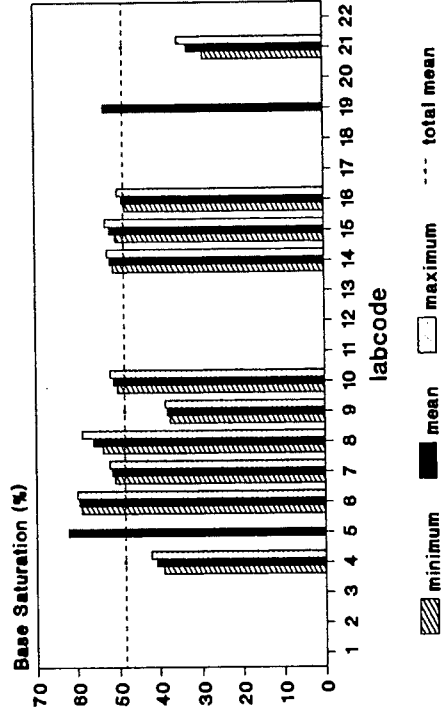


TABLE V : Base saturation Method comparison

General Linear Models Procedure Class Level Information			
Class	Levels	Values	
METH	6	1 11 12 3 7 8	
Number of observations in by group = 25			
NOTE: Due to missing values, only 22 observations can be used in this analysis.			
Dependent Variable: BASE SAT			
Source	DF	Sum of Squares	F Value Pr > F
Model	5	0.04313182	0.70 0.6324
Error	16	0.19760000	
Corrected Total	21	0.24073182	
		R-Square	C.V.
		0.179170	0.111180
		99.95590909	
Source	DF	Type I SS	F Value Pr > F
METH	5	0.04313182	0.70 0.6324
Source	DF	Type III SS	F Value Pr > F
METH	5	0.04313182	0.70 0.6324

TABLE IV : Base saturation - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	99.91	0.16	0.66	0.00
A1	3	1	99.93			
A1	7	3	100.00	0.00		
A1	8	5	100.00	0.00		
A1	11	3	100.00	0.00		
A1	12	1	99.91			
A2	3	1	90.72			0.86
A2	7	3	80.89	4.30	1.00	0.00
B1	1	34	38.01	7.28	0.89	0.82
B1	2	3	26.03	2.16	0.99	
B1	3	1	33.49			0.04
B1	4	7	29.61	2.18	0.80	0.33
B1	5	3	37.33	1.44	0.88	0.11
B1	7	3	28.04	0.68	0.80	0.74
B1	8	5	40.64	0.38	0.95	
B1	10	1	54.77			0.83
B1	11	3	86.76	0.54	0.99	0.02
B1	12	14	37.82	9.44	0.85	
B1	13	1	31.30			
B2	1	34	48.46	7.94	0.91	0.01
B2	2	3	8.10	0.87	0.84	0.22
B2	3	1	48.49			0.30
B2	4	7	40.36	3.99	0.89	0.16
B2	5	3	50.80	0.61	0.82	0.45
B2	7	3	44.66	0.52	0.92	0.74
B2	8	5	47.34	0.59	0.95	
B2	10	1	86.81			0.34
B2	11	3	80.19	2.69	0.89	0.00
B2	12	14	34.23	25.54	0.75	
B2	13	1	47.00			

Remark:
Labcode 3 reported mean values for 5 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for method 3 are different from the real number of replicates.

----- SAMPLE=A1 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: BASE SAT

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 16 MSE= 0.01235
Critical Value of Studentized Range= 4.557

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means	Simultaneous Upper Confidence Limit	
	Limit			Limit	
7 - 11	-0.2924		0.0000	0.2924	
7 - 8	-0.2615		0.0000	0.2615	
7 - 3	-0.3435		0.0700	0.4835	
7 - 1	-0.1487		0.0900	0.3287	
7 - 12	-0.3235		0.0900	0.5035	
11 - 7	-0.2924		0.0000	0.2924	
11 - 8	-0.2615		0.0000	0.2615	
11 - 3	-0.3435		0.0700	0.4835	
11 - 1	-0.1487		0.0900	0.3287	
11 - 12	-0.3235		0.0900	0.5035	
8 - 7	-0.2615		0.0000	0.2615	
8 - 11	-0.2615		0.0000	0.2615	
8 - 3	-0.3223		0.0700	0.4623	
8 - 1	-0.1097		0.0900	0.2897	
8 - 12	-0.3023		0.0900	0.4823	
3 - 7	-0.4835		-0.0700	0.3435	
3 - 11	-0.4835		-0.0700	0.3435	
3 - 8	-0.4623		-0.0700	0.3223	
3 - 1	-0.3574		0.0200	0.3974	
3 - 12	-0.4864		0.0200	0.5264	
1 - 7	-0.3287		-0.0900	0.1487	
1 - 11	-0.3287		-0.0900	0.1487	
1 - 8	-0.2897		-0.0900	0.1097	
1 - 3	-0.3974		-0.0200	0.3574	
1 - 12	-0.3774		0.0000	0.3774	
12 - 7	-0.5035		-0.0900	0.3235	
12 - 11	-0.5035		-0.0900	0.3235	
12 - 8	-0.4823		-0.0900	0.3023	
12 - 3	-0.5264		-0.0200	0.4864	
12 - 1	-0.3774		-0.0000	0.3774	

----- SAMPLE=A2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	3	1 3 7

Number of observations in by group = 10

NOTE: Due to missing values, only 4 observations can be used in this analysis.

Dependent Variable: BASE SAT

Source	DF	Sum of Squares	F Value	Pr > F
Model	1	72.52083333	3.93	0.1860
Error	2	36.92046667		
Corrected Total	3	109.44130000		
R-Square C.V. BASE SAT				
Mean		0.662646	5.155123	83.34500000

Source	DF	Type I SS	F Value	Pr > F
METH	1	72.52083333	3.93	0.1860
Source	DF	Type III SS	F Value	Pr > F
METH	1	72.52083333	3.93	0.1860

Tukey's Studentized Range (HSD) Test for variable: BASE SAT

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.

Calculation of the 0.05 quantile of the Studentized Range of
2 means with 2 error degrees of freedom failed.

----- SAMPLE=B1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	11	1 10 11 12 13 2 3 4 5 7 8

Number of observations in by group = 75

Dependent Variable: BASE SAT

Source	DF	Sum of Squares	F Value	Pr > F
Model	10	8716.68663182	18.91	0.0001
Error	64	2950.73960210		
Corrected Total	74	11667.42623392		
R-Square		C.V.		BASE SAT
Mean				

0.747096 17.64250 38.48712000

Source	DF	Type I SS	F Value	Pr > F
METH	10	8716.68663182	18.91	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	10	8716.68663182	18.91	0.0001

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: BASE SAT

NOTE: This test controls the type I experimentwise error rate.
Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 46.10531
Critical Value of Studentized Range= 4.720

Comparisons significant at the 0.05 level are indicated by '****'.

		Simultaneous		Simultaneous	
		Lower	Difference	Upper	
		Confidence	Between	Confidence	
		Limit	Means	Limit	
METH	Comparison				
11	- 10	5.813	31.983	58.152	***
11	- 8	29.566	46.117	62.668	***
11	- 1	35.093	48.743	62.392	***
11	- 12	34.516	48.935	63.353	***
11	- 5	30.919	49.423	67.928	***
11	- 3	27.097	53.267	79.436	***
11	- 13	29.287	55.457	81.626	***
11	- 4	41.505	57.144	72.783	***
11	- 7	40.212	58.717	77.221	***
11	- 2	42.219	60.723	79.228	***
10	- 11	-58.152	-31.983	-5.813	***
10	- 8	-10.693	14.134	38.961	
10	- 1	-6.234	16.760	39.754	
10	- 12	-6.507	16.952	40.411	
10	- 5	-8.729	17.441	43.610	
10	- 3	-10.767	21.284	53.335	
10	- 13	-8.577	23.474	55.525	
10	- 4	0.933	25.161	49.389	***
10	- 7	0.564	26.734	52.904	***
10	- 2	2.571	28.741	54.910	***
8	- 11	-62.668	-46.117	-29.566	***
8	- 10	-38.961	-14.134	10.693	
8	- 1	-8.229	2.626	13.481	
8	- 12	-8.990	2.818	14.625	
8	- 5	-13.244	3.307	19.858	
8	- 3	-17.677	7.150	31.977	
8	- 13	-15.487	9.340	34.167	
8	- 4	-2.243	11.027	24.298	
8	- 7	-3.951	12.600	29.151	
8	- 2	-1.944	14.607	31.158	
1	- 11	-62.392	-48.743	-35.093	***
1	- 10	-39.754	-16.760	6.234	
1	- 8	-13.481	-2.626	8.229	
1	- 12	-7.005	0.192	7.389	
1	- 5	-12.969	0.681	14.331	

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
13 - 7	-22.910	3.260	29.430			
13 - 2	-20.903	5.267	31.436			
4 - 11	-72.783	-57.144	-41.505			***
4 - 10	-49.389	-25.161	-0.933			***
4 - 8	-24.298	-11.027	2.243			
4 - 1	-17.808	-8.401	1.005			
4 - 12	-18.700	-8.209	2.282			
4 - 5	-23.360	-7.720	7.919			
4 - 3	-28.105	-3.877	20.351			
4 - 13	-25.915	-1.687	22.541			
4 - 7	-14.066	1.573	17.212			
4 - 2	-12.060	3.580	19.219			
7 - 11	-77.221	-58.717	-40.212			***
7 - 10	-52.904	-26.734	-0.564			***
7 - 8	-29.151	-12.600	3.951			
7 - 1	-23.624	-9.974	3.676			
7 - 12	-24.201	-9.782	4.637			
7 - 5	-27.798	-9.293	9.211			
7 - 3	-31.620	-5.450	20.720			
7 - 13	-29.430	-3.260	22.910			
7 - 4	-17.212	-1.573	14.066			
7 - 2	-16.498	2.007	20.511			
2 - 11	-79.228	-60.723	-42.219			***
2 - 10	-54.910	-28.741	-2.571			***
2 - 8	-31.158	-14.607	1.944			
2 - 1	-25.631	-11.981	1.669			
2 - 12	-26.208	-11.789	2.630			
2 - 5	-29.805	-11.300	7.205			
2 - 3	-33.626	-7.457	18.713			
2 - 13	-31.436	-5.267	20.903			
2 - 4	-19.219	-3.580	12.060			
2 - 7	-20.511	-2.007	16.498			

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 3	-18.470	4.524	27.518			
1 - 13	-16.280	6.714	29.708			
1 - 4	-1.005	8.401	17.808			
1 - 7	-3.676	9.974	23.624			
1 - 2	-1.669	11.981	25.631			
12 - 11	-63.353	-48.935	-34.516			***
12 - 10	-40.411	-16.952	6.507			
12 - 8	-14.625	-2.818	8.990			
12 - 1	-7.389	-0.192	7.005			
12 - 5	-13.930	0.489	14.908			
12 - 3	-19.127	4.332	27.791			
12 - 13	-16.937	6.522	29.981			
12 - 4	-2.282	8.209	18.700			
12 - 7	-4.637	9.782	24.201			
12 - 2	-2.630	11.789	26.208			
5 - 11	-67.928	-49.423	-30.919			***
5 - 10	-43.610	-17.441	8.729			
5 - 8	-19.858	-3.307	13.244			
5 - 1	-14.331	-0.681	12.969			
5 - 12	-14.908	-0.489	13.930			
5 - 3	-22.326	3.843	30.013			
5 - 13	-20.136	6.033	32.203			
5 - 4	-7.919	7.720	23.360			
5 - 7	-9.211	9.293	27.798			
5 - 2	-7.205	11.300	29.805			
3 - 11	-79.436	-53.267	-27.097			***
3 - 10	-53.335	-21.284	10.767			
3 - 8	-31.977	-7.150	17.677			
3 - 1	-27.518	-4.524	18.470			
3 - 12	-27.791	-4.332	19.127			
3 - 5	-30.013	-3.843	22.326			
3 - 13	-29.861	2.190	34.241			
3 - 4	-20.351	3.877	28.105			
3 - 7	-20.720	5.450	31.620			
3 - 2	-18.713	7.457	33.626			
13 - 11	-81.626	-55.457	-29.287			***
13 - 10	-55.525	-23.474	8.577			
13 - 8	-34.167	-9.340	15.487			
13 - 1	-29.708	-6.714	16.280			
13 - 12	-29.981	-6.522	16.937			
13 - 5	-32.203	-6.033	20.136			
13 - 3	-34.241	-2.190	29.861			
13 - 4	-22.541	1.687	25.915			

----- SAMPLE=B2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: BASE SAT

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 166.7269

Critical Value of Studentized Range= 4.720

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
10 - 11	-43.145	6.620	6.620	56.385	
10 - 5	-13.752	36.013	36.013	85.778	
10 - 3	-22.626	38.323	38.323	99.272	
10 - 1	-5.375	38.352	38.352	82.079	
10 - 8	-7.738	39.473	39.473	86.684	
10 - 13	-21.136	39.813	39.813	100.762	
10 - 7	-7.612	42.153	42.153	91.918	
10 - 4	0.378	46.452	46.452	92.525	***
10 - 12	7.971	52.581	52.581	97.191	***
10 - 2	28.948	78.713	78.713	128.478	***
11 - 10	-56.385	-6.620	-6.620	43.145	
11 - 5	-5.796	29.393	29.393	64.582	
11 - 3	-18.062	31.703	31.703	81.468	***
11 - 1	5.775	31.732	31.732	57.689	***
11 - 8	1.379	32.853	32.853	64.327	***
11 - 13	-16.572	33.193	33.193	82.958	
11 - 7	0.344	35.533	35.533	70.722	***
11 - 4	10.092	39.832	39.832	69.572	***
11 - 12	18.542	45.961	45.961	73.380	***
11 - 2	36.904	72.093	72.093	107.282	***
5 - 10	-85.778	-36.013	-36.013	13.752	
5 - 11	-64.582	-29.393	-29.393	5.796	
5 - 3	-47.455	2.310	2.310	52.075	
5 - 1	-23.618	2.339	2.339	28.296	
5 - 8	-28.014	3.460	3.460	34.934	
5 - 13	-45.965	3.800	3.800	53.565	
5 - 7	-29.049	6.140	6.140	41.329	
5 - 4	-19.302	10.439	10.439	40.179	
5 - 12	-10.851	16.568	16.568	43.987	***
5 - 2	7.511	42.700	42.700	77.889	***
3 - 10	-99.272	-38.323	-38.323	22.626	
3 - 11	-81.468	-31.703	-31.703	18.062	
3 - 5	-52.075	-2.310	-2.310	47.455	
3 - 1	-43.698	0.029	0.029	43.756	
3 - 8	-46.061	1.150	1.150	48.361	

----- SAMPLE=B2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	11	1 10 11 12 13 2 3 4 5 7 8

Number of observations in by group = 75

Dependent Variable: BASE SAT

Source	DF	Sum of Squares	F Value	Pr > F
Model	10	11874.5087425	7.12	0.0001
Error	64	10670.5194616		
Corrected Total	74	22545.0282041		
R-Square		C.V.		BASE SAT
Mean	0.526702	28.65393		45.06284000

Source	DF	Type I SS	F Value	Pr > F
METH	10	11874.5087425	7.12	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	10	11874.5087425	7.12	0.0001

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 12	-16.991	10.428	37.847	-16.991	10.428
7 - 2	1.371	36.560	71.749	1.371	36.560
4 - 10	-92.525	-46.452	-0.378	-92.525	-0.378
4 - 11	-69.572	-39.832	-10.092	-69.572	-10.092
4 - 5	-40.179	-10.439	19.302	-40.179	19.302
4 - 3	-54.202	-8.129	37.945	-54.202	37.945
4 - 1	-25.987	-8.099	9.788	-25.987	9.788
4 - 8	-32.214	-6.979	18.257	-32.214	18.257
4 - 13	-52.712	-6.639	39.435	-52.712	39.435
4 - 7	-34.039	-4.299	25.442	-34.039	25.442
4 - 12	-13.821	6.129	26.080	-13.821	26.080
4 - 2	2.521	32.261	62.002	2.521	62.002
12 - 10	-97.191	-52.581	-7.971	-97.191	-7.971
12 - 11	-73.380	-45.961	-18.542	-73.380	-18.542
12 - 5	-43.987	-16.568	10.851	-43.987	10.851
12 - 3	-58.868	-14.258	30.352	-58.868	30.352
12 - 1	-27.915	-14.229	-0.543	-27.915	-0.543
12 - 8	-35.561	-13.108	9.346	-35.561	9.346
12 - 13	-57.378	-12.768	31.842	-57.378	31.842
12 - 7	-37.847	-10.428	16.991	-37.847	16.991
12 - 4	-26.080	-6.129	13.821	-26.080	13.821
12 - 2	-1.287	26.132	53.551	-1.287	53.551
2 - 10	-128.478	-78.713	-28.948	-128.478	-28.948
2 - 11	-107.282	-72.093	-36.904	-107.282	-36.904
2 - 5	-77.889	-42.700	-7.511	-77.889	-7.511
2 - 3	-90.155	-40.390	9.375	-90.155	9.375
2 - 1	-66.318	-40.361	-14.404	-66.318	-14.404
2 - 8	-70.714	-39.240	-7.766	-70.714	-7.766
2 - 13	-88.665	-38.900	10.865	-88.665	10.865
2 - 7	-71.749	-36.560	-1.371	-71.749	-1.371
2 - 4	-62.002	-32.261	-2.521	-62.002	-2.521
2 - 12	-53.551	-26.132	1.287	-53.551	1.287

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
3 - 13	-59.459	62.439	1.490	-59.459	62.439
3 - 7	-45.935	53.595	3.830	-45.935	53.595
3 - 4	-37.945	54.202	8.129	-37.945	54.202
3 - 12	-30.352	58.868	14.258	-30.352	58.868
3 - 2	-9.375	90.155	40.390	-9.375	90.155
1 - 10	-82.079	5.375	-38.352	-82.079	5.375
1 - 11	-57.689	-5.775	-31.732	-57.689	-5.775
1 - 5	-28.296	23.618	-2.339	-28.296	23.618
1 - 3	-43.756	43.698	-0.029	-43.756	43.698
1 - 8	-19.522	21.763	1.121	-19.522	21.763
1 - 13	-42.266	45.188	1.461	-42.266	45.188
1 - 7	-22.156	29.758	3.801	-22.156	29.758
1 - 4	-9.788	25.987	8.099	-9.788	25.987
1 - 12	0.543	27.915	14.229	0.543	27.915
1 - 2	14.404	66.318	40.361	14.404	66.318
8 - 10	-86.684	7.738	-39.473	-86.684	7.738
8 - 11	-64.327	-1.379	-32.853	-64.327	-1.379
8 - 5	-34.934	-3.460	-3.460	-34.934	-3.460
8 - 3	-48.361	46.061	-1.150	-48.361	46.061
8 - 1	-21.763	19.522	-1.121	-21.763	19.522
8 - 13	-46.871	47.551	0.340	-46.871	47.551
8 - 7	-28.794	34.154	2.680	-28.794	34.154
8 - 4	-18.257	32.214	6.979	-18.257	32.214
8 - 12	-9.346	35.561	13.108	-9.346	35.561
8 - 2	7.766	70.714	39.240	7.766	70.714
13 - 10	-100.762	21.136	-39.813	-100.762	21.136
13 - 11	-82.958	16.572	-33.193	-82.958	16.572
13 - 5	-53.565	45.965	-3.800	-53.565	45.965
13 - 3	-62.439	59.459	-1.490	-62.439	59.459
13 - 1	-45.188	42.266	-1.461	-45.188	42.266
13 - 8	-47.551	46.871	-0.340	-47.551	46.871
13 - 7	-47.425	52.105	2.340	-47.425	52.105
13 - 4	-39.435	52.712	6.639	-39.435	52.712
13 - 12	-31.842	57.378	12.768	-31.842	57.378
13 - 2	-10.865	88.665	38.900	-10.865	88.665
7 - 10	-91.918	7.612	-42.153	-91.918	7.612
7 - 11	-70.722	-0.344	-35.533	-70.722	-0.344
7 - 5	-41.329	29.049	-6.140	-41.329	29.049
7 - 3	-53.595	45.935	-3.830	-53.595	45.935
7 - 1	-29.758	22.156	-3.801	-29.758	22.156
7 - 8	-34.154	28.794	-2.680	-34.154	28.794
7 - 13	-52.105	47.425	-2.340	-52.105	47.425
7 - 4	-25.442	34.039	4.299	-25.442	34.039

20. TOTAL N

units: mg/kg
mandatory for all samples
reported by 22 laboratories

TABLE I : Total N - Overview of reported data

labcode	samples reported	number of replicates	N/R method
1	A1 A2 B1 B2	3	N
2	A1 A2 B1 B2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	2	R
5	A1 A2 B1 B2	1 not reported	R
6	A1 A2 B1 B2	2	N
7	A1 A2 B1 B2	3	N/R
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	N
11	A1 A2 B1 B2	3	N
12	A1 A2 B1 B2	A1:8 A2:10 B1:7 B2	R
13	A1 A2 B1 B2	3	N
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	3	N
16	A1 A2 B1 B2	3	N
17	A1 A2 B1 B2	3	N
18	A1 A2 B1 B2	2	N
19	A1 A2 B1 B2	3	N=R
20	A1 A2 B1 B2	1 not reported	N=R
21	A1 A2 B1 B2	3	N
22	A1 A2 B1 B2	3	R

TABLE II : Total N - Summary method descriptions

method	description
1	reference
2	Kjeldahl
3	digestion with H2SO4, back titration with H3BO3
4	total ICP-AES analysis
5	wet ashed
6	AFNOR X31-111
7	thermal combustion
8	combustion on Carlo Erba
9	LECO CHN-600 analyser
10	unspecified

TABLE IIIa : Total N - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	2.30	2.37	2.40	0.06	2.44
2	3	3	2.07	2.31	2.46	0.21	9.09
3	4	5	2.63	2.77	2.89	0.09	3.25
4							
5							
6	5	2	2.02	2.07	2.12	0.07	3.42
7	6	3	2.46	2.52	2.57	0.05	1.88
8	10	3	1.71	1.83	1.98	0.14	7.51
9	2	3	2.20	2.20	2.20	0.00	0.00
10	2	3	2.21	2.23	2.25	0.02	0.94
11	2	3	2.47	2.49	2.51	0.02	0.84
12							
13	10	3	2.32	2.32	2.32	0.00	0.00
14	9	5	2.40	2.62	2.80	0.15	5.66
15	2	3	1.09	1.61	2.14	0.53	32.61
16	7	3	2.47	2.50	2.53	0.03	1.22
17	8	3	2.26	2.28	2.30	0.02	0.91
18							
19							
20	2	1		2.40			
21	2	3	1.30	1.36	1.41	0.06	4.09
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	15.70	15.83	15.90	0.12	0.73
2	3	3	18.62	18.67	18.78	0.09	0.50
3	4	5	20.95	23.06	25.10	1.86	8.15
4							
5							
6	5	2	17.88	18.01	18.14	0.18	1.02
7	6	3	17.70	17.90	18.00	0.17	0.97
8	10	3	22.96	23.38	23.77	0.41	1.74
9	2	3	16.10	16.37	16.50	0.23	1.41
10	2	3	16.10	16.23	16.40	0.15	0.94
11	2	3	14.89	15.52	15.90	0.47	3.05
12							
13	10	3	18.07	18.64	19.07	0.51	2.78
14	9	5	16.40	16.80	17.20	0.40	2.38
15	A3	5	16.20	16.30	16.40	0.10	0.61
16	2	3	17.77	17.93	18.18	0.22	1.22
17	7	3	18.60	18.98	18.99	0.10	0.52
18	8	3	16.41	16.77	17.00	0.32	1.90
19							
20	2	1		16.80			
21	2	3	6.32	7.32	8.86	1.35	18.45
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	0.30	0.33	0.40	0.06	17.32
2	3	3	0.28	0.32	0.34	0.03	10.15
3	4	5	0.23	0.24	0.24	0.00	0.00
4							
5							
6	5	2	0.08	0.08	0.08	0.00	0.00
7	6	3	0.29	0.30	0.32	0.02	5.77
8	10	3	0.15	0.18	0.20	0.03	14.25
9	2	3	0.20	0.27	0.30	0.06	21.65
10	2	3	0.24	0.26	0.27	0.02	6.86
11	2	3	0.31	0.32	0.32	0.01	1.82
12							
13	10	3	0.28	0.34	0.43	0.08	24.19
14	9	5	0.30	0.48	0.60	0.11	22.82
15	2	3	0.30	0.31	0.32	0.01	3.23
16	7	3	0.22	0.27	0.31	0.05	16.91
17	8	3	0.21	0.23	0.26	0.03	11.50
18							
19							
20	2	1		0.40			
21	2	3	0.17	0.17	0.18	0.01	3.33
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	2	3	2.70	3.03	3.20	0.29	9.52
2	3	3	3.00	3.07	3.13	0.07	2.14
3	4	5	4.08	5.33	7.28	1.18	22.14
4							
5							
6	5	2	2.39	2.44	2.49	0.07	2.90
7	6	3	3.35	3.45	3.53	0.09	2.63
8	10	3	3.05	3.26	3.48	0.22	6.60
9	2	3	3.00	3.07	3.10	0.06	1.88
10	2	3	2.80	2.84	2.89	0.05	1.67
11	2	3	2.73	2.95	3.12	0.20	6.74
12							
13	10	3	26.00	29.03	30.80	2.84	9.09
14	9	5	3.20	3.30	3.50	0.12	3.71
15	2	3	2.53	2.84	3.03	0.27	9.60
16	7	3	3.59	3.66	3.71	0.06	1.67
17	8	3	2.50	2.56	2.62	0.06	2.36
18							
19							
20	2	1		3.30			
21	2	3	1.97	2.06	2.20	0.12	5.98
22							

TABLE IIIb : Total N - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	2.19	2.20	2.20	0.01	0.32
5	1		2.38			
6						
7	3	2.25	2.30	2.38	0.06	2.39
8						
9						
10						
11						
12	8	2.43	2.53	2.59	0.05	2.04
13						
14						
15						
16						
17						
18	2	2.24	2.29	2.34	0.07	3.09
19	3		2.23			
20						
21						
22	3	2.70	2.83	3.00	0.15	5.39

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	15.52	16.74	17.96	1.73	10.31
5	1		16.67			
6						
7	3	18.00	18.57	18.90	0.49	2.66
8	3	22.96	23.38	23.77	0.41	1.74
9						
10						
11						
12	10	16.39	17.13	17.46	0.33	1.94
13						
14						
15						
16						
17						
18	2	16.74	16.85	16.96	0.16	0.92
19	3		15.02			
20						
21						
22	3	15.80	16.33	16.90	0.55	3.37

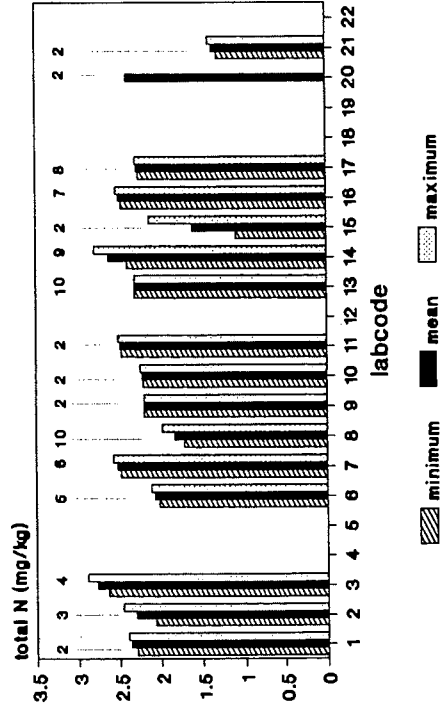
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	0.25	0.26	0.26	0.01	2.77
5	1		0.36			
6						
7	3	0.20	0.23	0.25	0.03	11.10
8						
9						
10						
11						
12	7	0.10	0.36	0.63	0.24	66.34
13						
14						
15						
16						
17						
18	2	0.20	0.25	0.30	0.07	28.28
19	3		0.28			
20						
21						
22	3	0.50	0.67	0.80	0.15	22.91

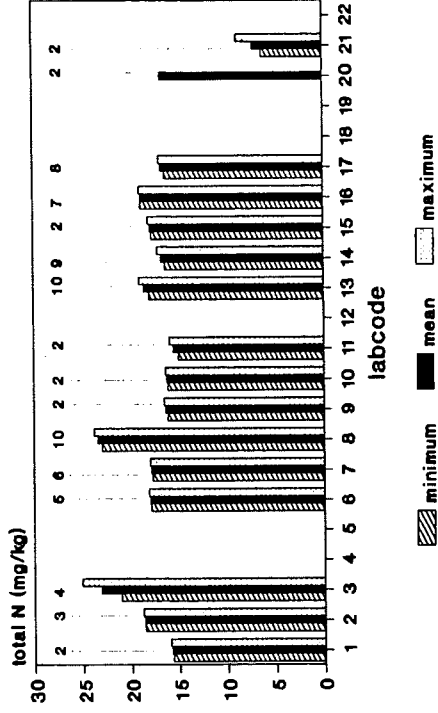
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4	2	2.15	2.32	2.49	0.24	10.36
5	1		2.36			
6						
7	3	3.54	3.73	3.93	0.20	5.23
8						
9						
10						
11						
12	9	2.75	2.96	3.13	0.16	5.37
13						
14						
15						
16						
17						
18	2	2.63	2.63	2.63	0.00	0.00
19	3		3.08			
20						
21						
22	3	3.50	3.77	3.90	0.23	6.13

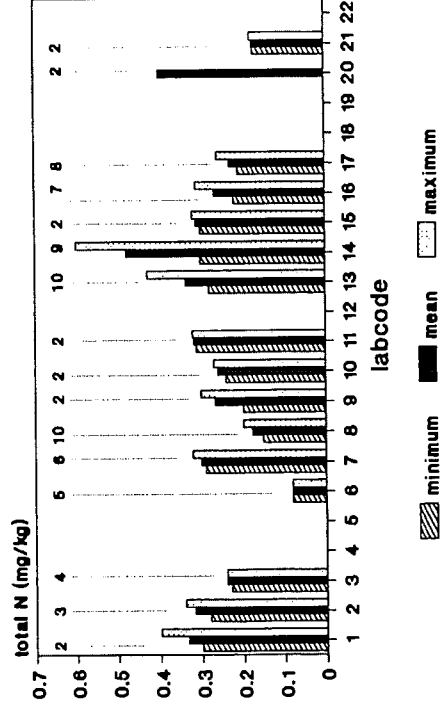
total N sample A1
national methods



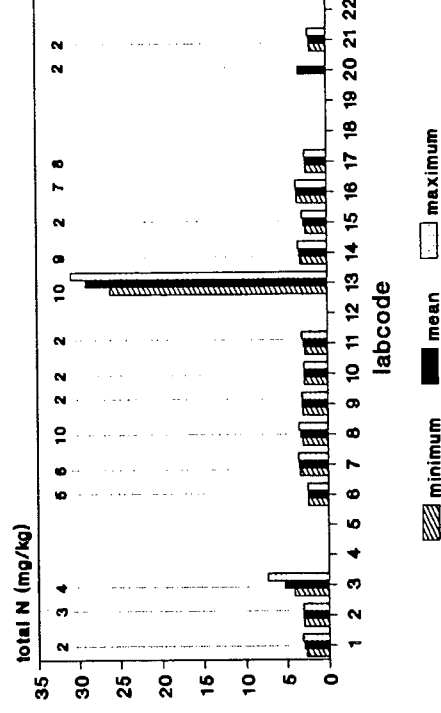
total N sample A2
national methods



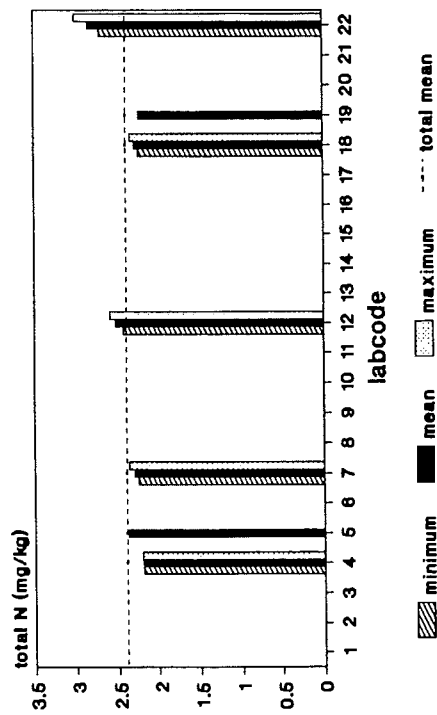
total N sample B1
national methods



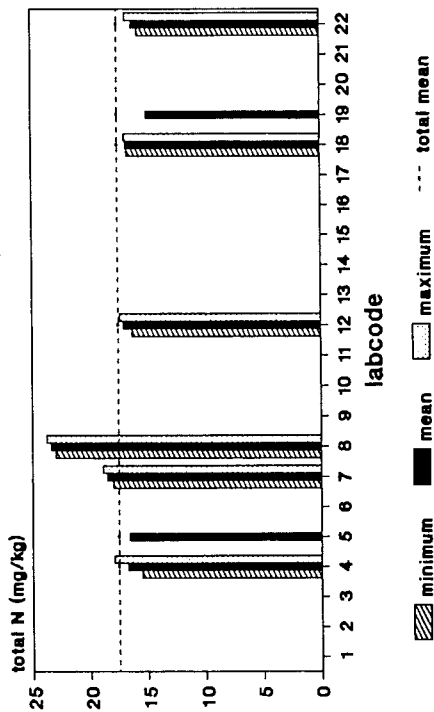
total N sample B2
national methods



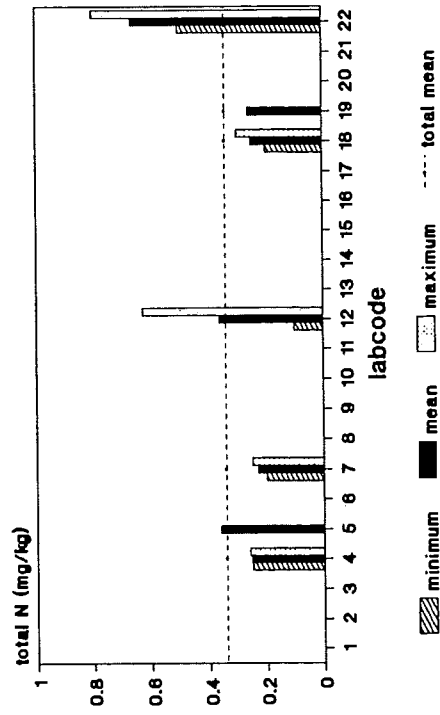
total N sample A1
reference method



total N sample A2
reference method



total N sample B1
reference method



total N sample B2
reference method

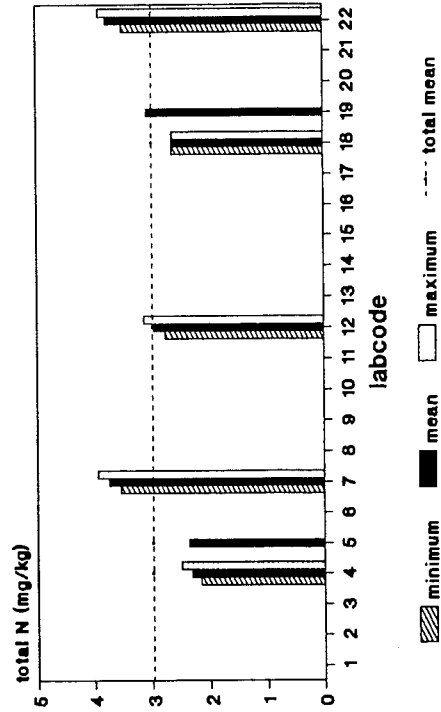


TABLE V : total N Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 2 3 4 5 6 7 8 9

Number of observations in by group = 65

Dependent Variable: TOTAL N

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	3.00179526	3.54	0.0016
Error	55	5.17664474		
Corrected Total	64	8.17844000		

R-Square	C.V.	TOTAL N Mean
0.367038	13.32716	2.30200000

Source	DF	Type I SS	F Value	Pr > F
METH	9	3.00179526	3.54	0.0016

Source	DF	Type III SS	F Value	Pr > F
METH	9	3.00179526	3.54	0.0016

TABLE IV : Total N - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	20	2.46	0.21	0.93	0.18
A1	2	19	2.06	0.46	0.80	0.00
A1	3	3	2.31	0.21	0.86	0.27
A1	4	1	2.77			
A1	5	2	2.07	0.07	1.00	1.00
A1	6	3	2.52	0.05	0.91	0.41
A1	7	3	2.50	0.03	0.96	0.64
A1	8	3	2.28	0.02	0.92	0.46
A1	9	5	2.62	0.15	0.96	0.78
A1	10	6	2.08	0.28	0.81	0.07
A2	1	25	17.80	2.28	0.75	0.00
A2	2	19	14.97	3.52	0.67	0.00
A2	3	3	18.67	0.09	0.75	0.00
A2	4	1	23.06			
A2	5	2	18.01	0.18	1.00	1.00
A2	6	3	17.90	0.17	0.75	0.00
A2	7	3	18.88	0.10	0.93	0.49
A2	8	3	16.77	0.32	0.86	0.27
A2	9	5	16.80	0.40	0.82	0.12
A2	10	6	21.01	2.63	0.81	0.07
A3	9	5	16.30	0.10	0.82	0.12
B1	1	19	0.36	0.21	0.87	0.01
B1	2	19	0.28	0.07	0.90	0.04
B1	3	3	0.32	0.03	0.87	0.30
B1	4	1	0.24			
B1	5	2	0.08	0.00		
B1	6	3	0.30	0.02	0.75	0.00
B1	7	3	0.27	0.05	1.00	0.88
B1	8	3	0.23	0.03	0.89	0.36
B1	9	5	0.48	0.11	0.83	0.13
B1	10	6	0.26	0.10	0.92	0.50
B2	1	21	3.07	0.52	0.95	0.33
B2	2	19	2.83	0.39	0.87	0.01
B2	3	3	3.07	0.07	0.98	0.75
B2	4	1	5.33			
B2	5	2	2.44	0.07	1.00	1.00
B2	6	3	3.45	0.09	0.98	0.76
B2	7	3	3.66	0.06	0.96	0.64
B2	8	3	2.56	0.06	0.99	0.82
B2	9	5	3.30	0.12	0.83	0.14
B2	10	6	16.15	14.22	0.74	0.02

Remark:
 Labcode 3 and labcode 19 reported mean values for respectively 3 and 5 replicates.
 To calculate the total number of replicates for the method-grouped laboratories,
 SAS counts the number of reported results.
 So, the "repl" values in this table for methods 4 and 1 are different from the real
 number of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: TOTAL N

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 55 MSE= 0.094121
Critical Value of Studentized Range= 4.662

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
4	- 9	-0.9579	0.1500	0.1500	1.2579	0.1500	1.2579
4	- 6	-0.9145	0.2533	0.2533	1.4212	0.2533	1.4212
4	- 7	-0.8945	0.2733	0.2733	1.4412	0.2733	1.4412
4	- 1	-0.7264	0.3100	0.3100	1.3464	0.3100	1.3464
4	- 3	-0.7079	0.4600	0.4600	1.6279	0.4600	1.6279
4	- 8	-0.6812	0.4867	0.4867	1.6545	0.4867	1.6545
4	- 10	-0.3974	0.6950	0.6950	1.7874	0.6950	1.7874
4	- 5	-0.5387	0.7000	0.7000	1.9387	0.7000	1.9387
4	- 2	-0.3282	0.7095	0.7095	1.7472	0.7095	1.7472
9	- 4	-1.2579	-0.1500	-0.1500	0.9579	-0.1500	0.9579
9	- 6	-0.6353	0.1033	0.1033	0.8420	0.1033	0.8420
9	- 7	-0.6153	0.1233	0.1233	0.8620	0.1233	0.8620
9	- 1	-0.3457	0.1600	0.1600	0.6657	0.1600	0.6657
9	- 3	-0.4286	0.3100	0.3100	1.0486	0.3100	1.0486
9	- 8	-0.4020	0.3367	0.3367	1.0753	0.3367	1.0753
9	- 10	-0.0674	0.5450	0.5450	1.1574	0.5450	1.1574
9	- 5	-0.2962	0.5500	0.5500	1.3962	0.5500	1.3962
9	- 2	0.0511	0.5595	0.5595	1.0678	0.5595	1.0678
6	- 4	-1.4212	-0.2533	-0.2533	0.9145	-0.2533	0.9145
6	- 9	-0.8420	-0.1033	-0.1033	0.6353	-0.1033	0.6353
6	- 7	-0.8058	0.0200	0.0200	0.8458	0.0200	0.8458
6	- 1	-0.5695	0.0567	0.0567	0.6829	0.0567	0.6829
6	- 3	-0.6191	0.2067	0.2067	1.0325	0.2067	1.0325
6	- 8	-0.5925	0.2333	0.2333	1.0591	0.2333	1.0591
6	- 10	-0.2735	0.4417	0.4417	1.1568	0.4417	1.1568
6	- 5	-0.4766	0.4467	0.4467	1.3700	0.4467	1.3700
6	- 2	-0.1722	0.4561	0.4561	1.0845	0.4561	1.0845
7	- 4	-1.4412	-0.2733	-0.2733	0.8945	-0.2733	0.8945
7	- 9	-0.8620	-0.1233	-0.1233	0.6153	-0.1233	0.6153
7	- 6	-0.8458	-0.0200	-0.0200	0.8058	-0.0200	0.8058
7	- 1	-0.5895	0.0367	0.0367	0.6629	0.0367	0.6629
7	- 3	-0.6391	0.1867	0.1867	1.0125	0.1867	1.0125
7	- 8	-0.6125	0.2133	0.2133	1.0391	0.2133	1.0391
7	- 10	-0.2935	0.4217	0.4217	1.1368	0.4217	1.1368
7	- 5	-0.4966	0.4267	0.4267	1.3500	0.4267	1.3500
7	- 2	-0.1922	0.4361	0.4361	1.0645	0.4361	1.0645
1	- 4	-1.3464	-0.3100	-0.3100	0.7264	-0.3100	0.7264
1	- 9	-0.6657	-0.1600	-0.1600	0.3457	-0.1600	0.3457
1	- 6	-0.6829	-0.0567	-0.0567	0.5695	-0.0567	0.5695
1	- 7	-0.6629	-0.0367	-0.0367	0.5895	-0.0367	0.5895
1	- 3	-0.4762	0.1500	0.1500	0.7762	0.1500	0.7762
1	- 8	-0.4495	0.1767	0.1767	0.8029	0.1767	0.8029
1	- 10	-0.0858	0.3850	0.3850	0.8558	0.3850	0.8558
1	- 5	-0.3601	0.3900	0.3900	1.1401	0.3900	1.1401
1	- 2	0.0755	0.3995	0.3995	0.7235	0.3995	0.7235
3	- 4	-1.6279	-0.4600	-0.4600	0.7079	-0.4600	0.7079
3	- 9	-1.0486	-0.3100	-0.3100	0.4286	-0.3100	0.4286
3	- 6	-1.0325	-0.2067	-0.2067	0.6191	-0.2067	0.6191
3	- 7	-1.0125	-0.1867	-0.1867	0.6391	-0.1867	0.6391
3	- 1	-0.7762	-0.1500	-0.1500	0.4762	-0.1500	0.4762
3	- 8	-0.7991	0.0267	0.0267	0.8525	0.0267	0.8525
3	- 10	-0.4802	0.2350	0.2350	0.9502	0.2350	0.9502
3	- 5	-0.6833	0.2400	0.2400	1.1633	0.2400	1.1633
3	- 2	-0.3789	0.2495	0.2495	0.8778	0.2495	0.8778
8	- 4	-1.6545	-0.4867	-0.4867	0.6812	-0.4867	0.6812
8	- 9	-1.0753	-0.3367	-0.3367	0.4020	-0.3367	0.4020
8	- 6	-1.0591	-0.2333	-0.2333	0.5925	-0.2333	0.5925
8	- 7	-1.0391	-0.2133	-0.2133	0.6125	-0.2133	0.6125
8	- 1	-0.8029	-0.1767	-0.1767	0.4495	-0.1767	0.4495
8	- 3	-0.8525	-0.0267	-0.0267	0.7991	-0.0267	0.7991
8	- 10	-0.5068	0.2083	0.2083	0.9235	0.2083	0.9235
8	- 5	-0.7100	0.2133	0.2133	1.1366	0.2133	1.1366
8	- 2	-0.4055	0.2228	0.2228	0.8512	0.2228	0.8512
10	- 4	-1.7874	-0.6950	-0.6950	0.3974	-0.6950	0.3974
10	- 9	-1.1574	-0.5450	-0.5450	0.0674	-0.5450	0.0674
10	- 6	-1.1568	-0.4417	-0.4417	0.2735	-0.4417	0.2735
10	- 7	-1.1368	-0.4217	-0.4217	0.2935	-0.4217	0.2935
10	- 1	-0.8558	-0.3850	-0.3850	0.0858	-0.3850	0.0858
10	- 3	-0.9502	-0.2350	-0.2350	0.4802	-0.2350	0.4802
10	- 8	-0.9235	-0.2083	-0.2083	0.5068	-0.2083	0.5068
10	- 5	-0.8208	0.0050	0.0050	0.8308	0.0050	0.8308
10	- 2	-0.4592	0.0145	0.0145	0.4881	0.0145	0.4881
5	- 4	-1.9387	-0.7000	-0.7000	0.5387	-0.7000	0.5387
5	- 9	-1.3962	-0.5500	-0.5500	0.2962	-0.5500	0.2962
5	- 6	-1.3700	-0.4467	-0.4467	0.4766	-0.4467	0.4766
5	- 7	-1.3500	-0.4267	-0.4267	0.4966	-0.4267	0.4966
5	- 1	-1.1401	-0.3900	-0.3900	0.3601	-0.3900	0.3601
5	- 3	-1.1633	-0.2400	-0.2400	0.6833	-0.2400	0.6833
5	- 8	-1.1366	-0.2133	-0.2133	0.7100	-0.2133	0.7100
5	- 10	-0.8308	-0.0050	-0.0050	0.8208	-0.0050	0.8208

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 2 3 4 5 6 7 8 9

Number of observations in by group = 70

Dependent Variable: TOTAL N

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	242.25118222	4.21	0.0003
Error	60	383.37141207		
Corrected Total	69	625.62259429		

R-Square	C.V.	TOTAL N Mean
0.387216	14.56054	17.36028571

Source	DF	Type I SS	F Value	Pr > F
METH	9	242.25118222	4.21	0.0003
Source	DF	Type III SS	F Value	Pr > F
METH	9	242.25118222	4.21	0.0003

SAMPLE=A1

General Linear Models Procedure

METH Comparison		Simultaneous			Simultaneous		
		Lower Confidence Limit	Difference Between Means	Upper Confidence Limit			
5	- 2	-0.7424	0.0095	0.7613			
2	- 4	-1.7472	-0.7095	0.3282			
2	- 9	-1.0678	-0.5595	-0.0511			***
2	- 6	-1.0845	-0.4561	0.1722			
2	- 7	-1.0645	-0.4361	0.1922			
2	- 1	-0.7235	-0.3995	-0.0755			***
2	- 3	-0.8778	-0.2495	0.3789			
2	- 8	-0.8512	-0.2228	0.4055			
2	- 10	-0.4881	-0.0145	0.4592			
2	- 5	-0.7613	-0.0095	0.7424			

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: TOTAL N

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 60 MSE= 6.389524
Critical Value of Studentized Range= 4.646

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous

Lower Confidence Limit

Difference Between Means

Upper Confidence Limit

METH Comparison

Simultaneous

Lower Confidence Limit

Difference Between Means

Upper Confidence Limit

Simultaneous

Lower Confidence Limit

Difference Between Means

Upper Confidence Limit

METH Comparison

Simultaneous

Lower Confidence Limit

Difference Between Means

Upper Confidence Limit

5	-	4	-15.221	-5.050	5.121
5	-	10	-9.781	-3.000	3.781
5	-	7	-8.451	-0.870	6.711
5	-	3	-8.245	-0.663	6.918
5	-	6	-7.471	0.110	7.691
5	-	1	-5.892	0.211	6.314
5	-	9	-5.738	1.210	8.158
5	-	8	-6.345	1.237	8.818
5	-	2	-3.133	3.041	9.214
6	-	4	-14.750	-5.160	4.430
6	-	10	-8.982	-3.110	2.762
6	-	7	-7.761	-0.980	5.801
6	-	3	-7.554	-0.773	6.007
6	-	5	-7.691	-0.110	7.471
6	-	1	-4.974	0.101	5.175
6	-	9	-4.965	1.100	7.165
6	-	8	-5.654	1.127	7.907
6	-	2	-2.229	2.931	8.090
1	-	4	-13.730	-5.261	3.208
1	-	10	-6.986	-3.211	0.565
1	-	7	-6.155	-1.081	3.994
1	-	3	-5.948	-0.874	4.200
1	-	5	-6.314	-0.211	5.892
1	-	6	-5.175	-0.101	4.974
1	-	9	-3.069	0.999	5.068
1	-	8	-4.048	1.026	6.100
1	-	2	0.302	2.830	5.357
9	-	4	-15.357	-6.260	2.837
9	-	10	-9.239	-4.210	0.819
9	-	7	-8.145	-2.080	3.985
9	-	3	-7.938	-1.873	4.192
9	-	5	-8.158	-1.210	5.738
9	-	6	-7.165	-1.100	4.965
9	-	1	-5.068	-0.999	3.069
9	-	8	-6.038	0.027	6.092
9	-	2	-2.344	1.831	6.005
8	-	4	-15.876	-6.287	3.303
8	-	10	-10.109	-4.237	1.636
8	-	7	-8.887	-2.107	4.674
8	-	3	-8.681	-1.900	4.881
8	-	5	-8.818	-1.237	6.345
8	-	6	-7.907	-1.127	5.654
8	-	1	-6.100	-1.026	4.048
8	-	9	-6.092	-0.027	6.038

4	-	10	-6.920	2.050	11.020
4	-	7	-5.410	4.180	13.770
4	-	3	-5.203	4.387	13.976
4	-	5	-5.121	5.050	15.221
4	-	6	-4.430	5.160	14.750
4	-	1	-3.208	5.261	13.730
4	-	9	-2.837	6.260	15.357
4	-	8	-3.303	6.287	15.876
4	-	2	-0.430	8.091	16.611
10	-	4	-11.020	-2.050	6.920
10	-	7	-3.742	2.130	8.002
10	-	3	-3.536	2.337	8.209
10	-	5	-3.781	3.000	9.781
10	-	6	-2.762	3.110	8.982
10	-	1	-0.565	3.211	6.986
10	-	9	-0.819	4.210	9.239
10	-	8	-1.636	4.237	10.109
10	-	2	2.151	6.041	9.930
7	-	4	-13.770	-4.180	5.410
7	-	10	-8.002	-2.130	3.742
7	-	3	-6.574	0.207	6.987
7	-	5	-6.711	0.870	8.451
7	-	6	-5.801	0.980	7.761
7	-	1	-3.994	1.081	6.155
7	-	9	-3.985	2.080	8.145
7	-	8	-4.674	2.107	8.887
7	-	2	-1.249	3.911	9.070
3	-	4	-13.976	-4.387	5.203
3	-	10	-8.209	-2.337	3.536
3	-	7	-6.987	-0.207	6.574
3	-	5	-6.918	0.663	8.245
3	-	6	-6.007	0.773	7.554
3	-	1	-4.200	0.874	5.948
3	-	9	-4.192	1.873	7.938
3	-	8	-4.881	1.900	8.681
3	-	2	-1.456	3.704	8.863

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	10	1 10 2 3 4 5 6 7 8 9

Number of observations in by group = 64

Dependent Variable: TOTAL N

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	0.36064386	2.20	0.0362
Error	54	0.98385614		
Corrected Total	63	1.34450000		

R-Square C.V. TOTAL N Mean

0.268236 43.36702 0.31125000

Source	DF	Type I SS	F Value	Pr > F
METH	9	0.36064386	2.20	0.0362
Source	DF	Type III SS	F Value	Pr > F
METH	9	0.36064386	2.20	0.0362

SAMPLE=A2

General Linear Models Procedure

METH Comparison		Simultaneous		Simultaneous	
		Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
8	- 2	-3.356	1.804	6.963	
2	- 4	-16.611	-8.091	0.430	***
2	- 10	-9.930	-6.041	-2.151	
2	- 7	-9.070	-3.911	1.249	
2	- 3	-8.863	-3.704	1.456	
2	- 5	-9.214	-3.041	3.133	
2	- 6	-8.090	-2.931	2.229	
2	- 1	-5.357	-2.830	-0.302	***
2	- 9	-6.005	-1.831	2.344	
2	- 8	-6.963	-1.804	3.356	

SAMPLE=BI

SAMPLE=BI

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: TOTAL N

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 54 MSE= 0.01822

Critical value of Studentized Range= 4.666

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
9 - 1	-0.1049	0.1189	0.3428	-0.4207	-0.1968	0.0270
9 - 3	-0.1619	0.1633	0.4886	-0.2224	-0.0779	0.0666
9 - 6	-0.1452	0.1800	0.5052	-0.3102	-0.0335	0.2432
9 - 2	-0.0270	0.1968	0.4207	-0.2935	-0.0168	0.2598
9 - 7	-0.1119	0.2133	0.5386	-0.2602	0.0165	0.2932
9 - 10	-0.0463	0.2233	0.4930	-0.1821	0.0265	0.2350
9 - 4	-0.2479	0.2400	0.7279	-0.4138	0.0432	0.5001
9 - 8	-0.0752	0.2500	0.5752	-0.2235	0.0532	0.3298
9 - 5	-0.0274	0.4000	0.7726	-0.1279	0.2032	0.5342
1 - 9	-0.3428	-0.1189	0.1049	-0.5386	-0.2133	0.1119
1 - 3	-0.2323	0.0444	0.3211	-0.3711	-0.0944	0.1823
1 - 6	-0.2156	0.0611	0.3377	-0.4136	-0.0500	0.3136
1 - 2	-0.0666	0.0779	0.2224	-0.3970	-0.0333	0.3303
1 - 7	-0.1823	0.0944	0.3711	-0.2932	-0.0165	0.2602
1 - 10	-0.1042	0.1044	0.3129	-0.3049	0.0100	0.3249
1 - 4	-0.3359	0.1211	0.5780	-0.4876	0.0267	0.5409
1 - 8	-0.1456	0.1311	0.4077	-0.3270	0.0367	0.4003
1 - 5	-0.0500	0.2811	0.6121	-0.2199	0.1867	0.5932
3 - 9	-0.4886	-0.1633	0.1619	-0.4930	-0.2233	0.0463
3 - 1	-0.3211	-0.0444	0.2323	-0.3129	-0.1044	0.1042
3 - 6	-0.3470	0.0167	0.3803	-0.3749	-0.0600	0.2549
3 - 2	-0.2432	0.0335	0.3102	-0.3582	-0.0433	0.2716
3 - 7	-0.3136	0.0500	0.4136	-0.2350	-0.0265	0.1821
3 - 10	-0.2549	0.0600	0.3749	-0.3249	-0.0100	0.3049
3 - 4	-0.4376	0.0767	0.5909	-0.4644	0.0167	0.4977
3 - 8	-0.2770	0.0867	0.4503	-0.2882	0.0267	0.3416
3 - 5	-0.1699	0.2367	0.6432	-0.1870	0.1767	0.5403
6 - 9	-0.5052	-0.1800	0.1452	-0.7279	-0.2400	0.2479
6 - 1	-0.3377	-0.0611	0.2156	-0.5780	-0.1211	0.3359
6 - 3	-0.3803	-0.0167	0.3470	-0.5909	-0.0767	0.4376
6 - 2	-0.2598	0.0168	0.2935	-0.5742	-0.0600	0.4542
6 - 7	-0.3303	0.0333	0.3970	-0.5001	-0.0432	0.4138
6 - 10	-0.2716	0.0433	0.3582	-0.5409	-0.0267	0.4876
6 - 4	-0.4542	0.0600	0.5742	-0.4977	-0.0167	0.4644
6 - 8	-0.2936	0.0700	0.4336	-0.5042	0.0100	0.5242
6 - 5	-0.1865	0.2200	0.6265	-0.3854	0.1600	0.7054
9 - 9	-0.5052	-0.1800	0.1452	-0.7279	-0.2400	0.2479
9 - 1	-0.3377	-0.0611	0.2156	-0.5780	-0.1211	0.3359
9 - 3	-0.3803	-0.0167	0.3470	-0.5909	-0.0767	0.4376
9 - 2	-0.2598	0.0168	0.2935	-0.5742	-0.0600	0.4542
9 - 7	-0.3303	0.0333	0.3970	-0.5001	-0.0432	0.4138
9 - 10	-0.2716	0.0433	0.3582	-0.5409	-0.0267	0.4876
9 - 4	-0.4542	0.0600	0.5742	-0.4977	-0.0167	0.4644
9 - 8	-0.2936	0.0700	0.4336	-0.5042	0.0100	0.5242
9 - 5	-0.1865	0.2200	0.6265	-0.3854	0.1600	0.7054
6 - 9	-0.5052	-0.1800	0.1452	-0.7279	-0.2400	0.2479
6 - 1	-0.3377	-0.0611	0.2156	-0.5780	-0.1211	0.3359
6 - 3	-0.3803	-0.0167	0.3470	-0.5909	-0.0767	0.4376
6 - 2	-0.2598	0.0168	0.2935	-0.5742	-0.0600	0.4542
6 - 7	-0.3303	0.0333	0.3970	-0.5001	-0.0432	0.4138
6 - 10	-0.2716	0.0433	0.3582	-0.5409	-0.0267	0.4876
6 - 4	-0.4542	0.0600	0.5742	-0.4977	-0.0167	0.4644
6 - 8	-0.2936	0.0700	0.4336	-0.5042	0.0100	0.5242
6 - 5	-0.1865	0.2200	0.6265	-0.3854	0.1600	0.7054
9 - 9	-0.5052	-0.1800	0.1452	-0.7279	-0.2400	0.2479
9 - 1	-0.3377	-0.0611	0.2156	-0.5780	-0.1211	0.3359
9 - 3	-0.3803	-0.0167	0.3470	-0.5909	-0.0767	0.4376
9 - 2	-0.2598	0.0168	0.2935	-0.5742	-0.0600	0.4542
9 - 7	-0.3303	0.0333	0.3970	-0.5001	-0.0432	0.4138
9 - 10	-0.2716	0.0433	0.3582	-0.5409	-0.0267	0.4876
9 - 4	-0.4542	0.0600	0.5742	-0.4977	-0.0167	0.4644
9 - 8	-0.2936	0.0700	0.4336	-0.5042	0.0100	0.5242
9 - 5	-0.1865	0.2200	0.6265	-0.3854	0.1600	0.7054

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 10 1 10 2 3 4 5 6 7 8 9

Number of observations in by group = 66

Dependent Variable: TOTAL N

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	945.11184493	5.77	0.0001
Error	56	1018.61233083		
Corrected Total	65	1963.72417576		

TOTAL N Mean

C.V.

R-Square

0.481285 100.5733 4.24060606

Source	DF	Type I SS	F Value	Pr > F
METH	9	945.11184493	5.77	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	945.11184493	5.77	0.0001

SAMPLE=B1

General Linear Models Procedure

Simultaneous Lower Confidence Limit

Simultaneous Upper Confidence Limit

Difference Between Means

METH Comparison

8	- 5	-0.2565	0.1500	0.5565	
5	- 9	-0.7726	-0.4000	-0.0274	***
5	- 1	-0.6121	-0.2811	0.0500	
5	- 3	-0.6432	-0.2367	0.1699	
5	- 6	-0.6265	-0.2200	0.1865	
5	- 2	-0.5342	-0.2032	0.1279	
5	- 7	-0.5932	-0.1867	0.2199	
5	- 10	-0.5403	-0.1767	0.1870	
5	- 4	-0.7054	-0.1600	0.3854	
5	- 8	-0.5565	-0.1500	0.2565	

SAMPLE=B2

General Linear Models Procedure

Simultaneous Lower Confidence Limit Difference Between Means Simultaneous Upper Confidence Limit

Tukey's Studentized Range (HSD) Test for variable: TOTAL N

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 56 MSE= 18.18951

Critical Value of Studentized Range= 4.659

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous Lower Confidence Limit Difference Between Means Simultaneous Upper Confidence Limit

METH Comparison

10 - 4 - 4.359 10.817 25.992 ***

10 - 7 - 2.555 12.490 22.425 ***

10 - 6 - 2.765 12.700 22.635 ***

10 - 9 - 4.339 12.847 21.354 ***

10 - 3 - 3.142 13.077 23.011 ***

10 - 1 - 6.575 13.079 19.582 ***

10 - 2 - 6.742 13.322 19.901 ***

10 - 8 - 3.655 13.590 23.525 ***

10 - 5 - 2.235 13.707 25.178 ***

4 - 10 - 25.992 -10.817 4.359

4 - 7 - -14.550 1.673 17.897

4 - 6 - -14.340 1.883 18.107

4 - 9 - -13.361 2.030 17.421

4 - 3 - -13.963 2.260 18.483

4 - 1 - -12.119 2.262 16.642

4 - 2 - -11.910 2.505 16.920

4 - 8 - -13.450 2.773 18.997

4 - 5 - -14.318 2.890 20.098

7 - 10 - -22.425 -12.490 -2.555 ***

7 - 4 - -17.897 -1.673 14.550

7 - 6 - -11.262 0.210 11.682

7 - 9 - -9.904 0.357 10.617

7 - 3 - -10.885 0.587 12.058

7 - 1 - -8.083 0.589 9.260

7 - 2 - -7.897 0.832 9.561

7 - 8 - -10.372 1.100 12.572

7 - 5 - -11.609 1.217 14.042

6 - 10 - -22.635 -12.700 -2.765 ***

6 - 4 - -18.107 -1.883 14.340

6 - 7 - -11.682 -0.210 11.262

6 - 9 - -10.114 0.147 10.407

6 - 3 - -11.095 0.377 11.848

6 - 1 - -8.293 0.379 9.050

6 - 2 - -8.107 0.622 9.351

6 - 8 - -10.582 0.890 12.362

6 - 5 - -11.819 1.007 13.832

SAMPLE=B2

General Linear Models Procedure

Simultaneous Lower Confidence Limit Difference Between Means Simultaneous Upper Confidence Limit

Tukey's Studentized Range (HSD) Test for variable: TOTAL N

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 56 MSE= 18.18951

Critical Value of Studentized Range= 4.659

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous Lower Confidence Limit Difference Between Means Simultaneous Upper Confidence Limit

METH Comparison

9 - 10 - -21.354 -12.847 -4.339 ***

9 - 4 - -17.421 -2.030 13.361

9 - 7 - -10.617 -0.357 9.904

9 - 6 - -10.407 -0.147 10.114

9 - 3 - -10.031 0.230 10.491

9 - 1 - -6.760 0.232 7.223

9 - 2 - -6.587 0.475 7.537

9 - 8 - -9.517 0.743 11.004

9 - 5 - -10.895 0.860 12.615

3 - 10 - -23.011 -13.077 -3.142 ***

3 - 4 - -18.483 -2.260 13.963

3 - 7 - -12.058 -0.587 10.885

3 - 6 - -11.848 -0.377 11.095

3 - 9 - -10.491 -0.230 10.031

3 - 1 - -8.670 0.002 8.674

3 - 2 - -8.483 0.245 8.974

3 - 8 - -10.958 0.513 11.985

3 - 5 - -12.196 0.630 13.456

1 - 10 - -19.582 -13.079 -6.575 ***

1 - 4 - -16.642 -2.262 12.119

1 - 7 - -9.260 -0.589 8.083

1 - 6 - -9.050 -0.379 8.293

1 - 9 - -7.223 -0.232 6.760

1 - 3 - -8.674 -0.002 8.670

1 - 2 - -4.205 0.243 4.692

1 - 8 - -8.160 0.511 9.183

1 - 5 - -9.769 0.628 11.025

2 - 10 - -19.901 -13.322 -6.742 ***

2 - 4 - -16.920 -2.505 11.910

2 - 7 - -9.561 -0.832 7.897

2 - 6 - -9.351 -0.622 8.107

2 - 9 - -7.537 -0.475 6.587

2 - 3 - -8.974 -0.245 8.483

2 - 1 - -4.692 -0.243 4.205

2 - 8 - -8.461 0.268 8.997

2 - 5 - -10.060 0.385 10.829

8 - 10 - -23.525 -13.590 -3.655 ***

8 - 4 - -18.997 -2.773 13.450

8 - 7 - -12.572 -1.100 10.372

8 - 9 - -12.362 -0.890 10.582

8 - 6 - -11.004 -0.743 9.517

8 - 3 - -11.985 -0.513 10.958

8 - 1 - -9.183 -0.511 8.160

8 - 2 - -8.997 -0.268 8.461

8 - 8 - -8.997 -0.268 8.461

----- SAMPLE=B2 -----

General Linear Models Procedure

Simultaneous

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
8 - 5	-12.709	0.117	12.942	
5 - 10	-25.178	-13.707	-2.235	***
5 - 4	-20.098	-2.890	14.318	
5 - 7	-14.042	-1.217	11.609	
5 - 6	-13.832	-1.007	11.819	
5 - 9	-12.615	-0.860	10.895	
5 - 3	-13.456	-0.630	12.196	
5 - 1	-11.025	-0.628	9.769	
5 - 2	-10.829	-0.385	10.060	
5 - 8	-12.942	-0.117	12.709	

21. P

units: mg/kg
mandatory for sample A2
reported by 21 laboratories

TABLE I : P - Overview of the reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N A2:N/R
2	A2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6	A1 A2 B1 B2	2	N A2:N/R
7	A1 A2 B1 B2	3	N A2:N/R
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11	A1 A2 B1 B2	3	R
12	A2	5	N
13	A2	3	N
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	4 A2:3	N A2:R
16	A1 A2 B1 B2	3	N A2:N/R
17	A2	3	N
18	A1 A2 B1 B2	3	N/R
19	A2	3	R
20	A2	1 not reported	N
21	A2	3	R
22	A2	3	R

(*) R = aqua regia

TABLE II : P - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	dry ashing
5	digestion, HNO3 under pressure
6	extractant HF
7	combustion, HClO4 + H2SO4
* 8	mineralisation
9	wet ashing
10	extractant 0.2N BaCl2
11	ignition, HF + HClO4
12	unspecified

* not applicable

TABLE IIIa : P - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	630.00	630.00	630.00	0.00	0.00
2	2	5	630.01	643.63	652.00	7.74	1.20
3	2	4	462.00	508.75	560.00	43.14	8.48
4	3	4					
5							
6	9	2	650.00	660.00	670.00	14.14	2.14
7	11	3	728.00	730.67	734.00	2.89	0.40
8	12	3	489.00	496.33	500.00	6.35	1.28
9	2	3	607.00	624.33	638.00	15.82	2.53
10							
11							
12							
13							
14	4	5	535.00	608.40	659.00	45.10	7.41
15	2	4	571.00	596.75	612.00	17.80	2.98
16	5	3	621.00	626.33	632.00	5.51	0.88
17							
18	3	3	451.10	454.48	460.25	5.01	1.10
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	1030.00	1050.00	1060.00	17.32	1.65
2	2	3	993.60	993.60	993.60	0.00	0.00
3	2	5	1011.60	1045.36	1069.83	21.68	2.07
4	3	4	1012.00	1033.25	1055.00	20.11	1.95
5							
6	9	2	880.00	890.00	900.00	14.14	1.59
7	11	3	1004.00	1024.33	1062.00	32.66	3.19
8	12	3	1800.00	2019.00	2240.00	220.01	10.90
9	2	3	901.00	941.00	988.00	43.92	4.67
10							
11							
12	10	5	481.00	496.60	503.00	9.02	1.82
13	12	3	222.00	225.00	229.00	3.61	1.60
14	4	5	980.00	1008.40	1029.00	20.31	2.01
15	A3	5	985.00	997.20	1012.00	11.65	1.17
16							
17	5	3	946.00	980.33	1039.00	51.05	5.21
18							
19	3	3	460.89	463.08	465.24	2.18	0.47
20							
21	6	1		724.00			
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	410.00	423.33	440.00	15.28	3.61
2							
3	2	5	340.40	345.05	349.07	3.72	1.08
4	3	4	282.00	286.25	294.00	5.44	1.90
5							
6	9	2	350.00	360.00	370.00	14.14	3.93
7	11	3	420.00	426.00	437.00	9.54	2.24
8	12	3	459.00	476.00	510.00	29.45	6.19
9	2	3	340.00	356.67	370.00	15.28	4.28
10							
11							
12							
13							
14	4	5	325.00	333.60	339.00	5.94	1.78
15	2	4	290.00	310.75	337.00	19.52	6.28
16	5	3	363.00	369.00	375.00	6.00	1.63
17							
18	3	3	315.67	322.43	328.86	6.60	2.05
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	160.00	176.67	200.00	20.82	11.78
2							
3	2	5	107.17	120.18	139.07	11.77	9.79
4	3	4	78.80	81.60	83.70	2.21	2.71
5							
6	9	2	90.00	90.00	90.00	0.00	0.00
7	11	3	159.00	167.67	172.00	7.51	4.48
8	12	3	305.00	335.33	376.00	36.61	10.92
9	2	3	100.00	113.33	120.00	11.55	10.19
10							
11							
12							
13							
14	4	5	126.00	129.80	134.00	3.03	2.34
15	2	4	71.40	80.05	85.60	6.07	7.58
16	5	3	123.00	131.67	138.00	7.77	5.90
17							
18	3	3	20.24	20.24	20.24	0.00	0.00
19							
20							
21							
22							

TABLE IIIb : P - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	489.00	496.67	501.00	6.68	1.34
11	3	580.00	610.00	650.00	36.06	5.91
12						
13						
14						
15						
16						
17						
18	3	580.14	627.55	694.94	59.95	9.55
19	3		608.00			
20						
21						
22						

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1	3	810.00	810.00	810.00	0.00	0.00
2						
3						
4						
5						
6						
7	3	982.00	1082.00	1171.00	94.98	8.78
8	3	1800.00	2019.00	2240.00	220.01	10.90
9						
10	3	518.00	535.00	551.00	16.52	3.09
11	3	880.00	906.67	940.00	30.55	3.37
12						
13						
14						
15	3	927.00	937.00	947.00	10.00	1.07
16	2	901.00	928.00	955.00	38.18	4.12
17	3	4260.00	5273.33	6460.00	1102.78	20.91
18	3	301.10	354.36	421.76	61.56	17.37
19	3		949.00			
20						
21	3	0.05	0.05	0.05	0.00	0.00
22	3	1466.00	1477.33	1495.00	15.50	1.05

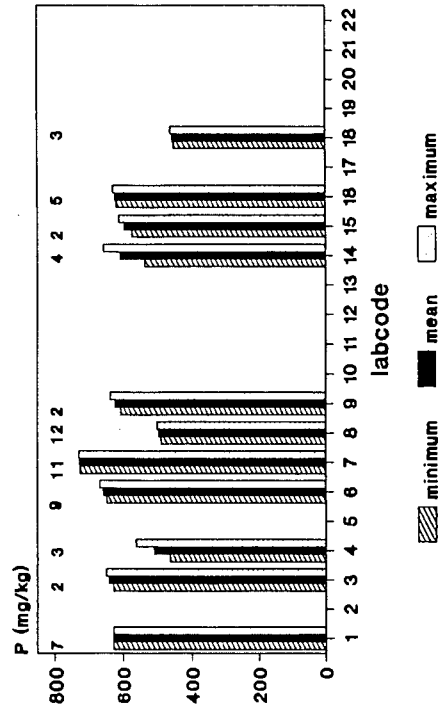
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	299.00	310.00	321.00	11.00	3.55
11	3	350.00	360.00	370.00	10.00	2.78
12						
13						
14						
15						
16						
17						
18	3	326.83	338.34	360.33	19.05	5.63
19	3		348.00			
20						
21						
22						

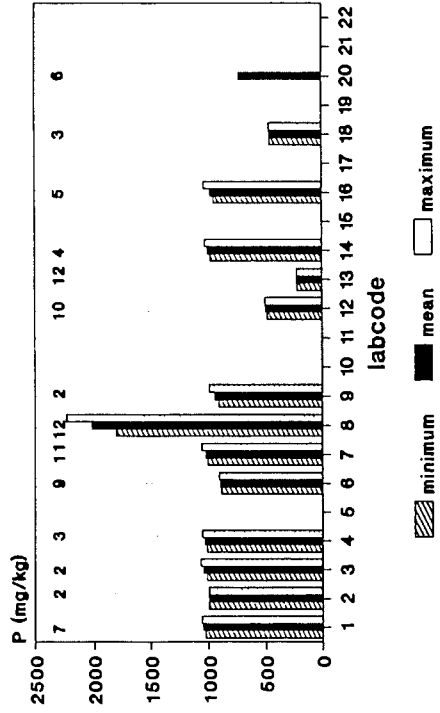
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	27.20	29.23	31.50	2.16	7.39
11	3	160.00	163.33	170.00	5.77	3.54
12						
13						
14						
15						
16						
17						
18	3	20.24	20.24	20.24	0.00	0.00
19	3		114.00			
20						
21						
22						

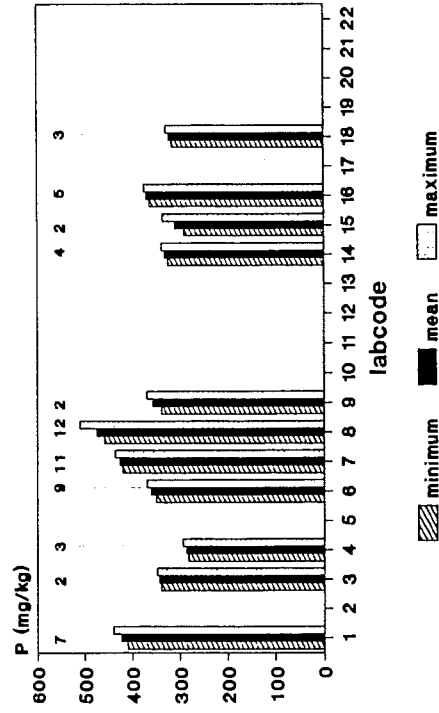
P sample A1
national methods



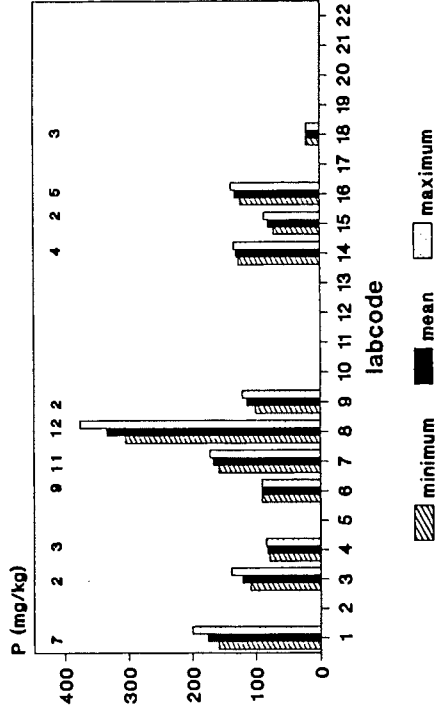
P sample A2
national methods



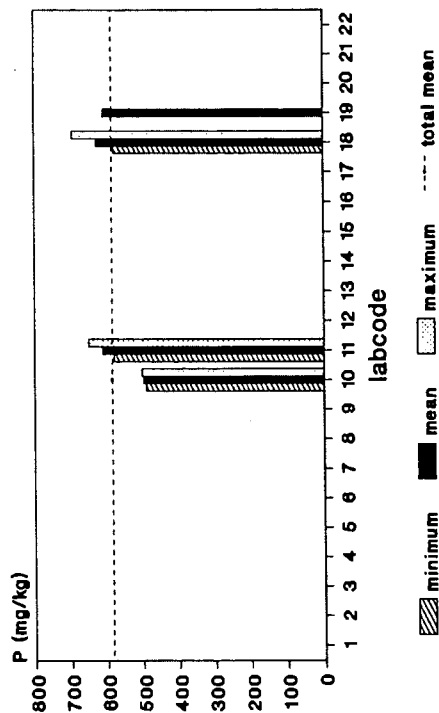
P sample B1
national methods



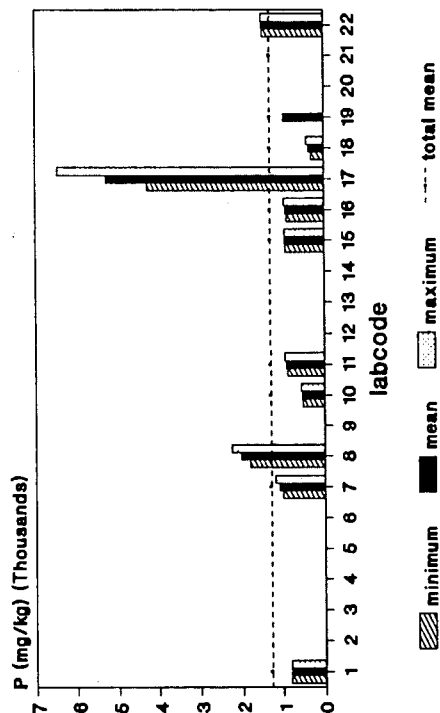
P sample B2
national methods



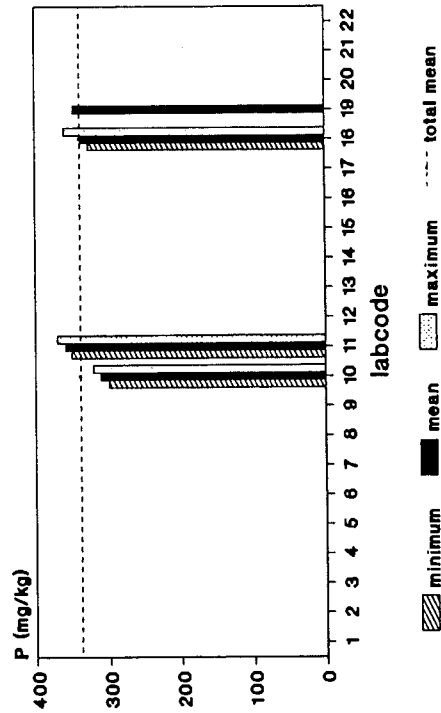
P sample A1
reference method



P sample A2
reference method



P sample B1
reference method



P sample B2
reference method

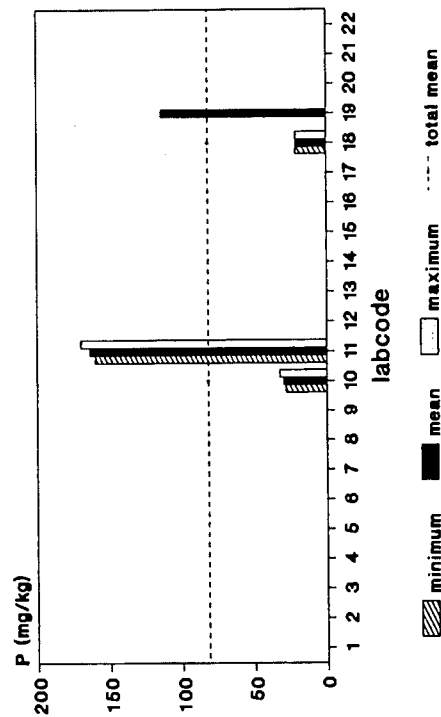


TABLE V : P Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	9	1 11 12 2 3 4 5 7 9

Number of observations in by group = 44

Dependent Variable: P				
Source	DF	Sum of Squares	F Value	Pr > F
Model	8	187367.919434	12.81	0.0001
Error	35	63970.512409		
Corrected Total	43	251338.431843		
R-Square				
C.V.				
P Mean				
7.255945				
589.19886364				
0.745481				
Type I SS				
F Value				
Pr > F				
Source	DF	Type I SS	F Value	Pr > F
METH	8	187367.919434	12.81	0.0001
Type III SS				
F Value				
Pr > F				
Source	DF	Type III SS	F Value	Pr > F
METH	8	187367.919434	12.81	0.0001

TABLE IV : P - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	10	581.07	67.47	0.93	0.40
A1	2	8	612.95	23.40	0.95	0.66
A1	3	7	485.50	42.19	0.83	0.07
A1	4	5	608.40	45.10	0.86	0.22
A1	5	3	626.33	5.51	1.00	0.90
A1	7	3	630.00	0.00		
A1	9	2	660.00	14.14	1.00	1.00
A1	11	3	730.67	2.89	0.75	0.00
A1	12	3	496.33	6.35	0.75	0.00
A2	1	33	1302.70	1405.31	0.69	0.00
A2	2	7	978.45	46.96	0.90	0.32
A2	3	7	788.88	305.11	0.69	0.00
A2	4	5	1008.40	20.31	0.94	0.66
A2	5	3	980.33	51.05	0.83	0.19
A2	6	1	724.00	17.32	0.75	0.00
A2	7	3	1050.00	14.14	1.00	1.00
A2	9	2	890.00	9.02	0.77	0.05
A2	10	5	496.60	32.66	0.79	0.09
A2	11	3	1024.33	992.42	0.76	0.03
A2	12	6	1122.00	11.65	0.92	0.50
A3	4	5	997.20	23.73	0.95	0.66
B1	1	10	337.30	27.80	0.95	0.75
B1	2	8	332.26	20.08	0.85	0.12
B1	3	7	301.76	5.94	0.89	0.37
B1	4	5	333.60	6.00	1.00	0.99
B1	5	3	369.00	15.28	0.96	0.64
B1	7	3	423.33	14.14	1.00	1.00
B1	9	2	360.00	9.54	0.79	0.10
B1	11	3	426.00	29.45	0.75	0.00
B1	12	3	476.00	66.90	0.75	0.00
B2	1	10	75.24	20.22	0.85	0.09
B2	2	8	97.55	32.84	0.69	0.00
B2	3	7	55.30	3.03	0.99	0.98
B2	4	5	129.80	7.77	0.93	0.50
B2	5	3	131.67	20.82	0.92	0.46
B2	7	3	176.67	0.00		
B2	9	2	90.00	7.51	0.75	0.00
B2	11	3	167.67	36.61	0.94	0.53
B2	12	3	335.33			

Remark:
Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for methods 2 and 1 are different from the real
number of replicates.

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: P

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 1827.729
Critical Value of Studentized Range= 4.671

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 9	-58.23	70.67	199.57			
11 - 7	-14.62	100.67	215.96			
11 - 5	-10.96	104.33	219.62			
11 - 2	22.12	117.71	213.31			
11 - 4	19.15	122.27	225.39			
11 - 1	56.65	149.60	242.55			
11 - 12	119.04	234.33	349.62			
11 - 3	147.73	245.17	342.61			
9 - 11	-199.57	-70.67	58.23			
9 - 7	-98.90	30.00	158.90			
9 - 5	-95.23	33.67	162.57			
9 - 2	-64.58	47.05	158.68			
9 - 4	-66.54	51.60	169.74			
9 - 1	-30.44	78.94	188.31			
9 - 12	34.77	163.67	292.57			
9 - 3	61.29	174.50	287.72			
7 - 11	-215.96	-100.67	14.62			
7 - 9	-158.90	-30.00	98.90			
7 - 5	-111.62	3.67	118.96			
7 - 2	-78.55	17.05	112.64			
7 - 4	-81.52	21.60	124.72			
7 - 1	-44.02	48.94	141.89			
7 - 12	18.38	133.67	248.96			
7 - 3	47.07	144.50	241.94			
5 - 11	-219.62	-104.33	10.96			
5 - 9	-162.57	-33.67	95.23			
5 - 7	-118.96	-3.67	111.62			
5 - 2	-82.22	13.38	108.97			
5 - 4	-85.19	17.93	121.05			
5 - 1	-47.68	45.27	138.22			
5 - 12	14.71	130.00	245.29			
5 - 3	43.40	140.84	238.28			
2 - 11	-213.31	-117.71	-22.12			
2 - 9	-158.68	-47.05	64.58			
2 - 7	-112.64	-17.05	78.55			

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
2 - 5	-108.97	-13.38	82.22			
2 - 4	-75.94	4.55	85.05			
2 - 1	-35.09	31.89	98.87			
2 - 12	21.03	116.62	212.22			
2 - 3	54.38	127.46	200.54			
4 - 11	-225.39	-122.27	-19.15			
4 - 9	-169.74	-51.60	66.54			
4 - 7	-124.72	-21.60	81.52			
4 - 5	-121.05	-17.93	85.19			
4 - 2	-85.05	-4.55	75.94			
4 - 1	-50.00	27.34	104.67			
4 - 12	8.95	112.07	215.19			
4 - 3	40.22	122.90	205.58			
1 - 11	-242.55	-149.60	-56.65			
1 - 9	-188.31	-78.94	30.44			
1 - 7	-141.89	-48.94	44.02			
1 - 5	-138.22	-45.27	47.68			
1 - 2	-98.87	-31.89	35.09			
1 - 4	-104.67	-27.34	50.00			
1 - 12	-8.22	84.73	177.68			
1 - 3	25.98	95.57	165.15			
12 - 11	-349.62	-234.33	-119.04			
12 - 9	-292.57	-163.67	-34.77			
12 - 7	-248.96	-133.67	-18.38			
12 - 5	-245.29	-130.00	-14.71			
12 - 2	-212.22	-116.62	-21.03			
12 - 4	-215.19	-112.07	-8.95			
12 - 1	-177.68	-84.73	8.22			
12 - 3	-86.60	10.84	108.28			
3 - 11	-342.61	-245.17	-147.73			
3 - 9	-287.72	-174.50	-61.29			
3 - 7	-241.94	-144.50	-47.07			
3 - 5	-238.28	-140.84	-43.40			
3 - 2	-200.54	-127.46	-54.38			
3 - 4	-205.58	-122.90	-40.22			
3 - 1	-165.15	-95.57	-25.98			
3 - 12	-108.28	-10.84	86.60			

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: P

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 1073485
Critical Value of Studentized Range= 4.720

Comparisons significant at the 0.05 level are indicated by '****'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1	- 12	-1354.1	180.7	1715.5	-1354.1	1715.5
1	- 7	-1832.7	252.7	2338.1	-1832.7	2338.1
1	- 11	-1807.0	278.4	2363.7	-1807.0	2363.7
1	- 4	-1365.3	294.3	1953.9	-1365.3	1953.9
1	- 5	-1763.0	322.4	2407.7	-1763.0	2407.7
1	- 2	-1114.8	324.3	1763.3	-1114.8	1763.3
1	- 9	-2105.6	412.7	2931.0	-2105.6	2931.0
1	- 3	-925.2	513.8	1952.9	-925.2	1952.9
1	- 6	-2931.5	578.7	4088.9	-2931.5	4088.9
1	- 10	-853.5	806.1	2465.7	-853.5	2465.7
12	- 1	-1715.5	-180.7	1354.1	-1715.5	1354.1
12	- 7	-2373.3	72.0	2517.3	-2373.3	2517.3
12	- 11	-2347.6	97.7	2543.0	-2347.6	2543.0
12	- 4	-1980.4	113.6	2207.6	-1980.4	2207.6
12	- 5	-2303.6	141.7	2587.0	-2303.6	2587.0
12	- 2	-1780.4	143.5	2067.5	-1780.4	2067.5
12	- 9	-2591.6	232.0	3055.6	-2591.6	3055.6
12	- 3	-1590.8	333.1	2257.1	-1590.8	2257.1
12	- 6	-3337.3	398.0	4133.3	-3337.3	4133.3
12	- 10	-1468.6	625.4	2719.4	-1468.6	2719.4
7	- 1	-2338.1	-252.7	1832.7	-2338.1	1832.7
7	- 12	-2517.3	-72.0	2373.3	-2517.3	2373.3
7	- 11	-2797.9	25.7	2849.3	-2797.9	2849.3
7	- 4	-2483.9	41.6	2567.1	-2483.9	2567.1
7	- 5	-2753.9	69.7	2893.3	-2753.9	2893.3
7	- 2	-2314.8	71.5	2457.9	-2314.8	2457.9
7	- 9	-2996.9	160.0	3316.9	-2996.9	3316.9
7	- 3	-2125.3	261.1	2647.5	-2125.3	2647.5
7	- 6	-3667.2	326.0	4319.2	-3667.2	4319.2
7	- 10	-1972.1	553.4	3078.9	-1972.1	3078.9
11	- 1	-2363.7	-278.4	1807.0	-2363.7	1807.0
11	- 12	-2543.0	-97.7	2347.6	-2543.0	2347.6
11	- 7	-2849.3	-25.7	2797.9	-2849.3	2797.9
11	- 4	-2509.6	15.9	2541.4	-2509.6	2541.4
11	- 5	-2779.6	44.0	2867.6	-2779.6	2867.6
11	- 2	-2340.5	45.9	2432.3	-2340.5	2432.3

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	11	1 10 11 12 2 3 4 5 6 7 9

Number of observations in by group = 75

Dependent Variable: P				
Source	DF	Sum of Squares	F Value	Pr > F
Model	10	4279770.00486	0.40	0.9425
Error	64	68703009.09740		
Corrected Total	74	72982779.10226		
C.V.				
R-Square			P Mean	
0.058641		95.59729	1083.8078667	
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	10	4279770.00486	0.40	0.9425
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	10	4279770.00486	0.40	0.9425

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
3 - 1	-1952.9	-513.8	925.2
3 - 12	-2257.1	-333.1	1590.8
3 - 7	-2647.5	-261.1	2125.3
3 - 11	-2621.8	-235.4	2150.9
3 - 4	-2244.4	-219.5	1805.4
3 - 5	-2577.8	-191.4	2194.9
3 - 2	-2038.0	-189.6	1658.9
3 - 9	-2873.8	-101.1	2671.6
3 - 6	-3632.1	64.9	3761.8
3 - 10	-1732.6	292.3	2317.2
6 - 1	-4088.9	-578.7	2931.5
6 - 12	-4133.3	-398.0	3337.3
6 - 7	-4319.2	-326.0	3667.2
6 - 11	-4293.5	-300.3	3692.8
6 - 4	-4072.7	-284.4	3503.9
6 - 5	-4249.5	-256.3	3736.8
6 - 2	-3951.4	-254.5	3442.5
6 - 9	-4401.4	-166.0	4069.4
6 - 3	-3761.8	-64.9	3632.1
6 - 10	-3560.9	227.4	4015.7
10 - 1	-2465.7	-806.1	853.5
10 - 12	-2719.4	-625.4	1468.6
10 - 7	-3078.9	-553.4	1972.1
10 - 11	-3053.2	-527.7	1997.8
10 - 4	-2699.0	-511.8	1675.4
10 - 5	-3009.2	-483.7	2041.8
10 - 2	-2506.8	-481.9	1543.1
10 - 9	-3286.7	-393.4	2499.9
10 - 3	-2317.2	-292.3	1732.6
10 - 6	-4015.7	-227.4	3560.9

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
11 - 9	-3022.5	134.3	3291.2
11 - 3	-2150.9	235.4	2621.8
11 - 6	-3692.8	300.3	4293.5
11 - 10	-1997.8	527.7	3053.2
4 - 1	-1953.9	-294.3	1365.3
4 - 12	-2207.6	-113.6	1980.4
4 - 7	-2567.1	-41.6	2483.9
4 - 11	-2541.4	-15.9	2509.6
4 - 5	-2497.4	28.1	2553.6
4 - 2	-1995.0	29.9	2054.9
4 - 9	-2774.9	118.4	3011.7
4 - 3	-1805.4	219.5	2244.4
4 - 6	-3503.9	284.4	4072.7
4 - 10	-1675.4	511.8	2699.0
5 - 1	-2407.7	-322.4	1763.0
5 - 12	-2587.0	-141.7	2303.6
5 - 7	-2893.3	-69.7	2753.9
5 - 11	-2867.6	-44.0	2779.6
5 - 4	-2553.6	-28.1	2497.4
5 - 2	-2384.5	1.9	2388.3
5 - 9	-3066.5	90.3	3247.2
5 - 3	-2194.9	191.4	2577.8
5 - 6	-3736.8	256.3	4249.5
5 - 10	-2041.8	483.7	3009.2
2 - 1	-1763.3	-324.3	1114.8
2 - 12	-2067.5	-143.5	1780.4
2 - 7	-2457.9	-71.5	2314.8
2 - 11	-2432.3	-45.9	2340.5
2 - 4	-2054.9	-29.9	1995.0
2 - 5	-2388.3	-1.9	2384.5
2 - 9	-2684.3	88.5	2861.2
2 - 3	-1658.9	189.6	2038.0
2 - 6	-3442.5	254.5	3951.4
2 - 10	-1543.1	481.9	2506.8
9 - 1	-2931.0	-412.7	2105.6
9 - 12	-3055.6	-232.0	2591.6
9 - 7	-3316.9	-160.0	2996.9
9 - 11	-3291.2	-134.3	3022.5
9 - 4	-3011.7	-118.4	2774.9
9 - 5	-3247.2	-90.3	3066.5
9 - 2	-2861.2	-88.5	2684.3
9 - 3	-2671.6	101.1	2873.8
9 - 6	-4069.4	166.0	4401.4
9 - 10	-2499.9	393.4	3286.7

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure
Class Level Information

Tukey's Studentized Range (HSD) Test for variable: P

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 448.4139
Critical Value of Studentized Range= 4.671

Comparisons significant at the 0.05 level are indicated by '****'.

NOTE: Due to missing values, only 44 observations can be used in this analysis.

Dependent Variable: P

Source	DF	Sum of Squares	F Value	Pr > F
Model	8	103095.794568	28.74	0.0001
Error	35	15694.486687		
Corrected Total	43	118790.281255		

R-Square	C.V.	P Mean
0.867881	5.967165	354.87181818

Source	DF	Type I SS	F Value	Pr > F
METH	8	103095.794568	28.74	0.0001

Source	DF	Type III SS	F Value	Pr > F
METH	8	103095.794568	28.74	0.0001

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Limit	Confidence Limit	Between Means	Upper Limit	Lower Limit	Upper Limit
12 - 11	-7.106		50.000		-7.106	107.106
12 - 7	-4.439		52.667		-4.439	109.773
12 - 5	49.894		107.000		49.894	164.106
12 - 9	52.154		116.000		52.154	179.846
12 - 1	92.659		138.699		92.659	184.739
12 - 4	91.323		142.400		91.323	193.477
12 - 2	96.394		143.744		96.394	191.094
12 - 3	125.980		174.243		125.980	222.506
11 - 12	-107.106		-50.000		-107.106	7.106
11 - 7	-54.439		2.667		-54.439	59.773
11 - 5	-0.106		57.000		-0.106	114.106
11 - 9	2.154		66.000		2.154	129.846
11 - 1	42.659		88.699		42.659	134.739
11 - 4	41.323		92.400		41.323	143.477
11 - 2	46.394		93.744		46.394	141.094
11 - 3	75.980		124.243		75.980	172.506
7 - 12	-109.773		-52.667		-109.773	4.439
7 - 11	-59.773		-2.667		-59.773	54.439
7 - 5	-2.773		54.333		-2.773	111.439
7 - 9	-0.513		63.333		-0.513	127.180
7 - 1	39.992		86.032		39.992	132.073
7 - 4	38.656		89.733		38.656	140.810
7 - 2	43.727		91.077		43.727	138.427
7 - 3	73.313		121.576		73.313	169.840
5 - 12	-164.106		-107.000		-164.106	-49.894
5 - 11	-114.106		-57.000		-114.106	0.106
5 - 7	-111.439		-54.333		-111.439	2.773
5 - 9	-54.846		9.000		-54.846	72.846
5 - 1	-14.341		31.699		-14.341	77.739
5 - 4	-15.677		35.400		-15.677	86.477
5 - 2	-10.606		36.744		-10.606	84.094
5 - 3	18.980		67.243		18.980	115.506
9 - 12	-179.846		-116.000		-179.846	-52.154
9 - 11	-129.846		-66.000		-129.846	-2.154
9 - 7	-127.180		-63.333		-127.180	0.513

SAMPLE=B2

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	9	1 11 12 2 3 4 5 7 9

Number of observations in by group = 45

NOTE: Due to missing values, only 44 observations can be used in this analysis.

Dependent Variable: P

Source	DF	Sum of Squares	F Value	Pr > F
Model	8	211395.085299	17.31	0.0001
Error	35	53432.528920		
Corrected Total	43	264827.614218		

R-Square C.V. P Mean

0.798237 33.17012 117.79363636

Source	DF	Type I SS	F Value	Pr > F
METH	8	211395.085299	17.31	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	8	211395.085299	17.31	0.0001

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	9	5	9	5	9	5
9	-72.846	-9.000	54.846	***	76.874	***
9	-31.476	22.699	76.874	***	84.916	***
9	-32.116	26.400	84.916	***	83.036	***
9	-27.549	27.744	83.036	***	114.320	***
9	2.166	58.243	114.320	***		
1	-184.739	-138.699	-92.659	***	-92.659	***
1	-134.739	-88.699	-42.659	***	-42.659	***
1	-132.073	-86.032	-39.992	***	14.341	***
1	-77.739	-31.699	31.476	***	42.009	***
1	-76.874	-22.699	31.476	***	38.220	***
1	-34.607	3.701	42.009	***	70.011	***
1	-28.131	5.045	38.220	***		
1	1.077	35.544	70.011	***		
4	-193.477	-142.400	-91.323	***	-91.323	***
4	-143.477	-92.400	-41.323	***	-41.323	***
4	-140.810	-89.733	-38.656	***	15.677	***
4	-86.477	-35.400	32.116	***	34.607	***
4	-84.916	-26.400	32.116	***	41.216	***
4	-42.009	-3.701	41.216	***	72.796	***
4	-38.528	1.344	41.216	***		
4	-9.110	31.843	72.796	***		
2	-191.094	-143.744	-96.394	***	-96.394	***
2	-141.094	-93.744	-46.394	***	-46.394	***
2	-138.427	-91.077	-43.727	***	10.606	***
2	-84.094	-36.744	27.549	***	28.131	***
2	-83.036	-27.744	27.549	***	38.528	***
2	-38.220	-5.045	28.131	***	66.697	***
2	-41.216	-1.344	38.528	***		
2	-5.698	30.499	66.697	***		
3	-222.506	-174.243	-125.980	***	-125.980	***
3	-172.506	-124.243	-75.980	***	-75.980	***
3	-169.840	-121.576	-73.313	***	-18.980	***
3	-115.506	-67.243	-18.980	***	-2.166	***
3	-114.320	-58.243	-2.166	***	-1.077	***
3	-70.011	-35.544	-1.077	***	9.110	***
3	-72.796	-31.843	9.110	***	5.698	***
3	-66.697	-30.499	5.698	***		

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: P

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 1526.644
Critical Value of Studentized Range= 4.671

Comparisons significant at the 0.05 level are indicated by '***'.

		METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
4	- 5	4	- 5	-96.11	-1.87	92.38	***		
4	- 2	4	- 2	-41.32	32.25	105.82			
4	- 9	4	- 9	-68.17	39.80	147.77			
4	- 1	4	- 1	-16.13	54.56	125.24			
4	- 3	4	- 3	-1.07	74.50	150.06			
2	- 12	2	- 12	-325.15	-237.79	-150.42	***		
2	- 7	2	- 7	-166.49	-79.12	8.25			
2	- 11	2	- 11	-157.49	-70.12	17.25			
2	- 5	2	- 5	-121.49	-34.12	53.25			
2	- 4	2	- 4	-105.82	-32.25	41.32			
2	- 9	2	- 9	-94.48	7.55	109.57			
2	- 1	2	- 1	-38.91	22.31	83.52			
2	- 3	2	- 3	-24.54	42.24	109.03			
9	- 12	9	- 12	-363.14	-245.33	-127.53	***		
9	- 7	9	- 7	-204.47	-86.67	31.14			
9	- 11	9	- 11	-195.47	-77.67	40.14			
9	- 5	9	- 5	-159.47	-41.67	76.14			
9	- 4	9	- 4	-147.77	-39.80	68.17			
9	- 2	9	- 2	-109.57	-7.55	94.48			
9	- 1	9	- 1	-85.20	14.76	114.72			
9	- 3	9	- 3	-68.77	34.70	138.17			
1	- 12	1	- 12	-345.04	-260.09	-175.14	***		
1	- 7	1	- 7	-186.38	-101.42	-16.47	***		
1	- 11	1	- 11	-177.38	-92.42	-7.47	***		
1	- 5	1	- 5	-141.38	-56.42	28.53			
1	- 4	1	- 4	-125.24	-54.56	16.13			
1	- 2	1	- 2	-83.52	-22.31	38.91			
1	- 9	1	- 9	-114.72	-14.76	85.20			
1	- 3	1	- 3	-43.66	19.94	83.54			
3	- 12	3	- 12	-369.08	-280.03	-190.98	***		
3	- 7	3	- 7	-210.42	-121.36	-32.31	***		
3	- 11	3	- 11	-201.42	-112.36	-23.31	***		
3	- 5	3	- 5	-165.42	-76.36	12.69			
3	- 4	3	- 4	-150.06	-74.50	1.07			
3	- 2	3	- 2	-109.03	-42.24	24.54			
3	- 9	3	- 9	-138.17	-34.70	68.77			
3	- 1	3	- 1	-83.54	-19.94	43.66			

		METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
12	- 7	12	- 7	53.30	158.67	264.04	***		
12	- 11	12	- 11	62.30	167.67	273.04	***		
12	- 5	12	- 5	98.30	203.67	309.04	***		
12	- 4	12	- 4	111.29	205.53	299.78	***		
12	- 2	12	- 2	150.42	237.79	325.15	***		
12	- 9	12	- 9	127.53	245.33	363.14	***		
12	- 1	12	- 1	175.14	260.09	345.04	***		
12	- 3	12	- 3	190.98	280.03	369.08	***		
7	- 12	7	- 12	-264.04	-158.67	-53.30	***		
7	- 11	7	- 11	-96.37	9.00	114.37			
7	- 5	7	- 5	-60.37	45.00	150.37			
7	- 4	7	- 4	-47.38	46.87	141.11			
7	- 2	7	- 2	-8.25	79.12	166.49			
7	- 9	7	- 9	-31.14	86.67	204.47	***		
7	- 1	7	- 1	16.47	101.42	186.38	***		
7	- 3	7	- 3	32.31	121.36	210.42	***		
11	- 12	11	- 12	-273.04	-167.67	-62.30	***		
11	- 7	11	- 7	-114.37	-9.00	96.37			
11	- 5	11	- 5	-69.37	36.00	141.37			
11	- 4	11	- 4	-56.38	37.87	132.11			
11	- 2	11	- 2	-17.25	70.12	157.49			
11	- 9	11	- 9	-40.14	77.67	195.47	***		
11	- 1	11	- 1	7.47	92.42	177.38	***		
11	- 3	11	- 3	23.31	112.36	201.42	***		
5	- 12	5	- 12	-309.04	-203.67	-98.30	***		
5	- 7	5	- 7	-150.37	-45.00	60.37			
5	- 11	5	- 11	-141.37	-36.00	69.37			
5	- 4	5	- 4	-92.38	1.87	96.11			
5	- 2	5	- 2	-53.25	34.12	121.49			
5	- 9	5	- 9	-76.14	41.67	159.47			
5	- 1	5	- 1	-28.53	56.42	141.38			
5	- 3	5	- 3	-12.69	76.36	165.42			
4	- 12	4	- 12	-299.78	-205.53	-111.29	***		
4	- 7	4	- 7	-141.11	-46.87	47.38			
4	- 11	4	- 11	-132.11	-37.87	56.38			

22. K

units: mg/kg
mandatory for sample A2
reported by 21 laboratories

TABLE I : K - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N A2:N/R
2	A2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5	A2	1 not reported	N
6	A1 A2 B1 B2	2	N
7	A1 A2 B1 B2	3	N A2:N/R
8	A1 A2 B1 B2	3	N A2:N/R
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11	A1 A2 B1	3	R
12	A2	5	N
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	4 A2:3	N
16	A1 A2 B1 B2	3	R A2:N/R
17	A2	3	R
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20	A2	1 not reported	N
21	A2	3	R
22	A2	3	R

(*) R = aqua regia

TABLE II : K - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	dry ashing
5	digestion, HNO3 under pressure
6	extractant HF
7	combustion, HClO4 + H2SO4
* 8	minéralisation
9	wet ashing
10	extractant 0.2N BaCl2 at pH 8.1
11	ignition, HF + HClO4
12	unspecified

* not applicable

TABLE IIIb : K - Total mean and normal distribution

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	1530.00	1607.33	1695.00	82.98	5.16
11	3	1250.00	1310.00	1380.00	65.57	5.01
12						
13						
14						
15						
16	3	2530.00	2566.67	2620.00	47.26	1.84
17						
18	3	2195.58	2279.23	2324.61	72.53	3.18
19	3		970.00			
20						
21						
22						

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1	3	3000.00	3200.00	3400.00	200.00	6.25
2						
3						
4						
5						
6						
7	3	2644.00	2918.00	3175.00	265.91	9.11
8	3	1255.00	1323.00	1413.00	81.27	6.14
9						
10	3	1801.00	1826.67	1863.00	32.35	1.77
11	3	1800.00	1900.00	1970.00	88.88	4.68
12						
13						
14						
15						
16	3	1930.00	2233.33	2460.00	273.19	12.23
17	3	2450.00	2653.33	3050.00	343.56	12.95
18	3	2440.32	2499.02	2594.67	83.55	3.34
19	3		2237.00			
20						
21	3	1670.00	1670.00	1670.00	0.00	0.00
22	3	3401.00	3422.67	3463.00	34.96	1.02

sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	2652.00	2694.33	2726.00	38.14	1.42
11	3	2980.00	3043.33	3100.00	60.28	1.98
12						
13						
14						
15						
16	3	3620.00	3626.67	3640.00	11.55	0.32
17						
18	3	3654.00	3658.74	3665.17	5.77	0.16
19	3		1373.00			
20						
21						
22						

sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	266.00	270.00	272.00	3.46	1.28
11	3	200.00	220.00	240.00	20.00	9.09
12						
13						
14						
15						
16	3	417.00	440.33	463.00	23.01	5.23
17						
18	3	423.02	436.51	444.27	11.73	2.69
19	3		280.00			
20						
21						
22						

TABLE IIIa : K - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	2500.00	2500.00	2500.00	0.00	0.00
2							
3	2	5	2514.80	2598.86	2730.23	84.06	3.23
4	3	4	689.00	772.50	865.00	79.10	10.24
5							
6	9	2	12550.00	12600.00	12650.00	70.71	0.56
7	11	3	16432.00	16585.00	16687.00	134.93	0.81
8	12	3	6349.00	6349.00	6349.00	0.00	0.00
9	2	3	1350.00	1406.67	1520.00	98.15	6.98
10							
11							
12							
13							
14	4	5	730.00	939.00	1066.00	125.64	13.38
15	2	4	951.00	1049.00	1102.00	71.38	6.81
16							
17							
18	3	3	985.52	995.00	1010.92	13.87	1.39
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	2600.00	2833.33	3100.00	251.86	8.88
2	2	3	3036.00	3220.00	3312.00	159.35	4.95
3	2	5	2982.00	3230.30	3462.15	218.56	6.77
4	3	4	1519.00	1553.50	1601.00	34.35	2.21
5	12	1	1445.00	1445.00	1445.00		
6	9	2	3700.00	3800.00	3900.00	141.42	3.72
7	11	3	5127.00	5916.00	6841.00	865.06	14.62
8	12	3	2947.00	3046.00	3207.00	140.65	4.62
9	2	3	2200.00	2240.00	2260.00	34.84	1.55
10							
11							
12	9	5	1736.00	1787.60	1860.00	64.49	3.61
13							
14	4	5	1643.00	1740.00	1842.00	88.51	5.09
14	A3	5	1635.00	1709.20	1786.00	64.03	3.75
15	2	3	112.30	131.80	154.60	21.47	16.29
16	5	3	3980.00	4106.67	4330.00	193.99	4.72
17							
18	3	3	1581.59	1589.20	1597.89	8.20	0.52
19							
20	6	1		4110.00			
21							
22							

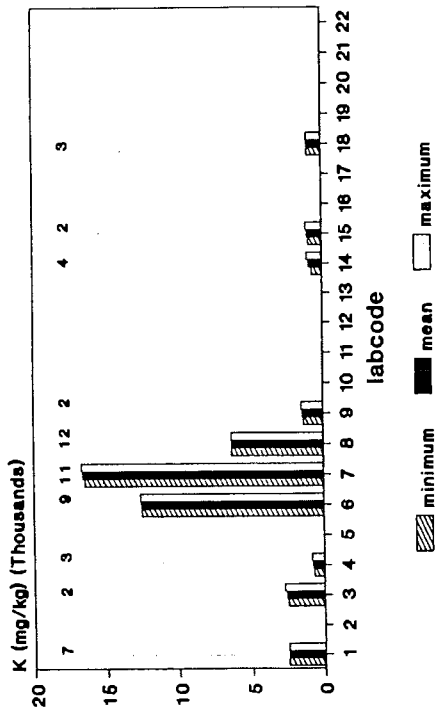
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	3800.00	3800.00	3800.00	0.00	0.00
2							
3	2	5	3558.34	3737.80	3975.12	159.13	4.26
4	3	4	1194.00	1221.00	1274.00	35.98	2.95
5							
6	9	2	7600.00	7700.00	7800.00	141.42	1.84
7	11	3	18910.00	18988.67	19115.00	110.50	0.58
8	12	3	5079.00	5319.00	5629.00	281.80	5.29
9	2	3	2440.00	2646.67	2880.00	221.21	8.36
10							
11							
12							
13							
14	4	5	2377.00	2600.40	2749.00	143.61	5.52
15	2	4	1787.00	2268.75	2887.00	514.70	22.69
16							
17							
18	3	3	1880.80	1898.36	1911.25	15.24	0.80
19							
20							
21							
22							

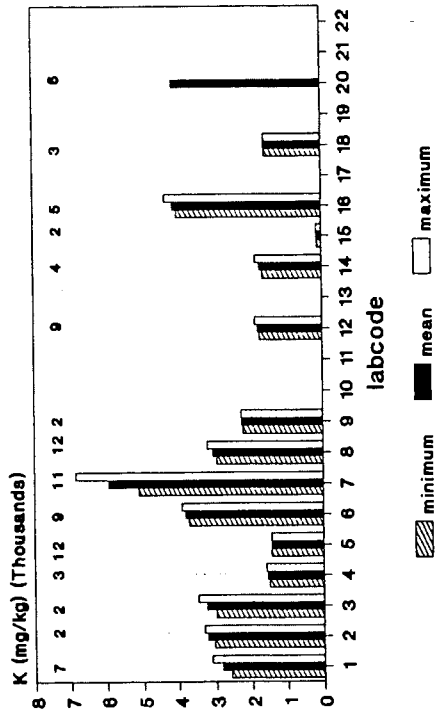
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	500.00	500.00	500.00	0.00	0.00
2							
3	2	5	496.94	526.26	563.02	21.90	4.16
4	3	4	242.00	252.25	267.00	10.81	4.29
5							
6	9	2	1000.00	1000.00	1000.00	0.00	0.00
7	11	3	4927.00	5130.67	5263.00	179.00	3.49
8	12	3	844.00	858.00	866.00	24.25	2.83
9	2	3	330.00	333.33	340.00	5.77	1.73
10							
11							
12							
13							
14	4	5	372.00	390.40	405.00	12.42	3.18
15	2	4	175.40	193.65	214.10	15.87	8.19
16							
17							
18	3	3	300.56	319.79	331.94	16.85	5.27
19							
20							
21							
22							

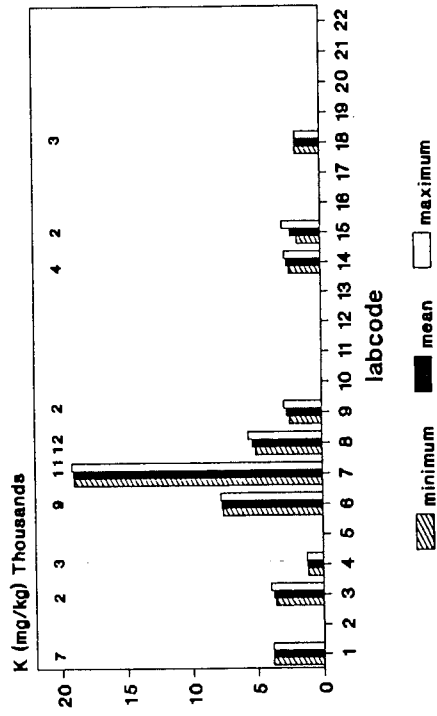
K sample A1
national methods



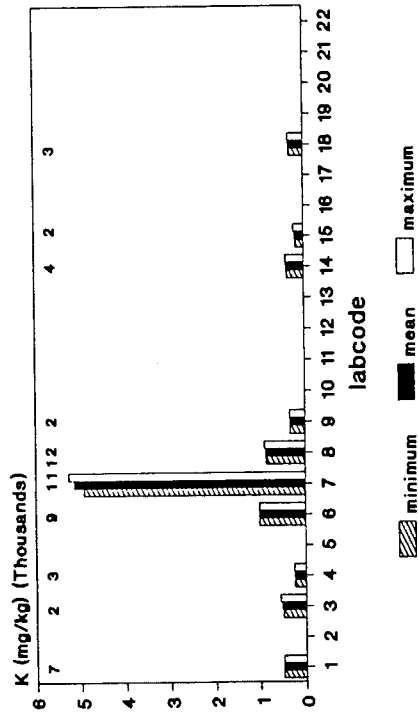
K sample A2
national methods



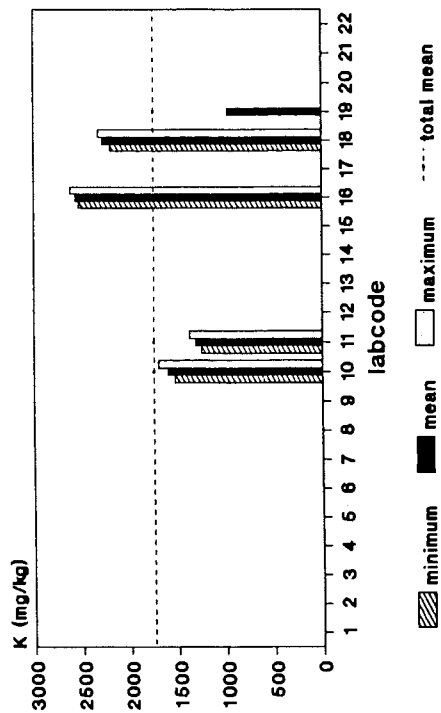
K sample B1
national methods



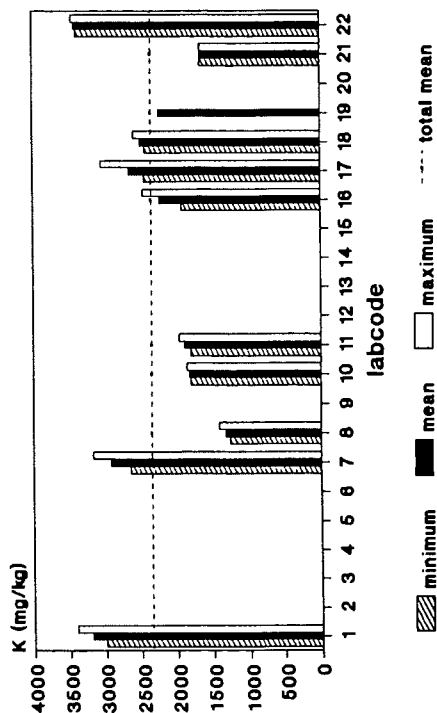
K sample B2
national methods



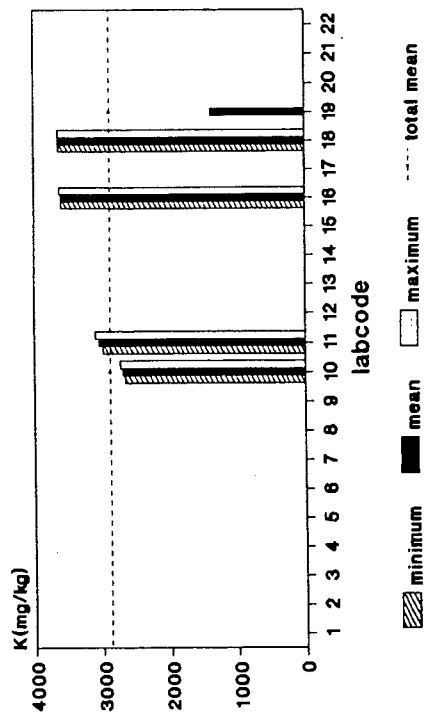
K sample A1
reference method



K sample A2
reference method



K sample B1
reference method



K sample B2
reference method

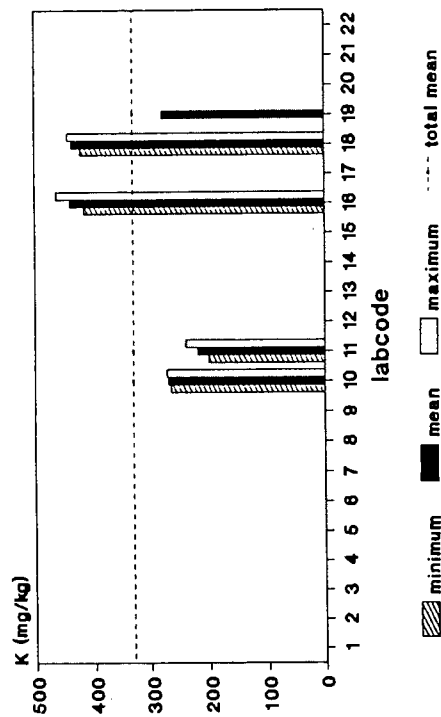


TABLE V : K Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 11 12 2 3 4 7 9

Number of observations in by group = 44

Dependent Variable: K

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	857732120.224	720.50	0.0001
Error	36	6122438.898		
Corrected Total	43	863854559.122		

R-Square C.V. K Mean

0.992913 12.29800 3353.3309091

Source	DF	Type I SS	F Value	Pr > F
METH	7	857732120.224	720.50	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	7	857732120.224	720.50	0.0001

TABLE IV : K - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	1866.13	573.98	0.91	0.18
A1	2	8	1376.86	529.21	0.74	0.01
A1	3	7	867.86	131.67	0.89	0.27
A1	4	5	939.00	125.64	0.87	0.26
A1	7	3	2500.00	0.00		
A1	9	2	12600.00	70.71	1.00	1.00
A1	11	3	16585.00	134.93	0.89	0.36
A1	12	3	6349.00	0.00		
A2	1	31	2360.49	673.67	0.94	0.08
A2	2	10	2000.57	1361.22	0.80	0.02
A2	3	7	1568.80	31.25	0.90	0.33
A2	4	5	1740.00	88.51	0.87	0.27
A2	5	3	4106.67	193.99	0.81	0.15
A2	6	1	4110.00			
A2	7	3	2833.33	251.66	0.99	0.78
A2	9	7	2362.57	985.06	0.66	0.00
A2	11	3	5916.00	865.06	0.98	0.07
A2	12	4	2645.75	808.70	0.76	0.04
A3	4	5	1709.20	64.03	0.78	0.06
B1	1	13	3110.94	662.29	0.80	0.01
B1	2	8	2594.10	613.23	0.94	0.63
B1	3	7	1510.44	362.00	0.71	0.00
B1	4	5	2600.40	143.61	0.94	0.64
B1	7	3	3800.00	0.00		
B1	9	2	7700.00	141.42	1.00	1.00
B1	11	3	18988.60	110.50	0.86	0.27
B1	12	3	5319.00	281.60	0.95	0.59
B2	1	13	336.96	100.70	0.84	0.02
B2	2	8	287.61	119.14	0.85	0.09
B2	3	7	281.20	38.17	0.86	0.16
B2	4	5	390.40	12.42	0.98	0.92
B2	7	3	500.00	0.00		
B2	9	2	1000.00	0.00		
B2	11	3	5130.67	179.00	0.88	0.33
B2	12	3	858.00	24.25	0.75	

Remark:

Labcode 3 and labcode 19 reported mean values for resp. 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for methods 2 and 1 are different from the real number of replicates.

K Method comparison

K Method comparison

SAMPLE=Al

SAMPLE=Al

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 170067.7
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 9	2774.5	3985.0	5195.5	-16105.9	-15208.1	-14310.4
11 - 12	9153.3	10236.0	11318.7	-12271.5	-11223.1	-10174.8
11 - 7	13002.3	14085.0	15167.7	-5869.9	-4972.1	-4074.4
11 - 1	13869.5	14718.9	15568.2	-2020.9	-1123.1	-225.4
11 - 2	14310.4	15208.1	16105.9	-1085.1	-489.3	106.6
11 - 4	14677.6	15646.0	16614.4	-318.1	437.9	1193.8
11 - 3	14802.1	15717.1	16632.2	-177.3	509.0	1195.3
9 - 11	-5195.5	-3985.0	-2774.5	-16614.4	-15646.0	-14677.6
9 - 12	5040.5	6251.0	7461.5	-12770.4	-11661.0	-10551.6
9 - 7	8889.5	10100.0	11310.5	-6378.4	-5410.0	-4441.6
9 - 1	9726.7	10733.9	11741.1	-2529.4	-1561.0	-592.6
9 - 2	10174.8	11223.1	12271.5	-1624.9	-927.1	-229.3
9 - 4	10551.6	11661.0	12770.4	-1193.8	-437.9	318.1
9 - 3	10668.9	11732.1	12795.3	-705.3	71.1	847.6
12 - 11	-11318.7	-10236.0	-9153.3	-16632.2	-15717.1	-14802.1
12 - 9	-7461.5	-6251.0	-5040.5	-12795.3	-11732.1	-10668.9
12 - 7	2766.3	3849.0	4931.7	-6396.2	-5481.1	-4566.1
12 - 1	3633.5	4482.9	5332.2	-2547.2	-1632.1	-717.1
12 - 2	4074.4	4972.1	5869.9	-1619.9	-998.3	-376.6
12 - 4	4441.6	5410.0	6378.4	-1195.3	-509.0	177.3
12 - 3	4566.1	5481.1	6396.2	-847.6	-71.1	705.3
7 - 11	-15167.7	-14085.0	-13002.3			
7 - 9	-11310.5	-10100.0	-8889.5			
7 - 12	-4931.7	-3849.0	-2766.3			
7 - 1	-215.5	633.9	1483.2			
7 - 2	225.4	1123.1	2020.9			
7 - 4	592.6	1561.0	2529.4			
7 - 3	717.1	1632.1	2547.2			
1 - 11	-15568.2	-14718.9	-13869.5			
1 - 9	-11741.1	-10733.9	-9726.7			
1 - 12	-5332.2	-4482.9	-3633.5			
1 - 7	-1483.2	-633.9	215.5			
1 - 2	-106.6	489.3	1085.1			
1 - 4	229.3	927.1	1624.9			
1 - 3	376.6	998.3	1619.9			

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 170067.7

Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
2 - 11	-16105.9	-15208.1	-14310.4	-16105.9	-15208.1	-14310.4
2 - 9	-12271.5	-11223.1	-10174.8	-12271.5	-11223.1	-10174.8
2 - 12	-5869.9	-4972.1	-4074.4	-5869.9	-4972.1	-4074.4
2 - 7	-2020.9	-1123.1	-225.4	-2020.9	-1123.1	-225.4
2 - 1	-1085.1	-489.3	106.6	-1085.1	-489.3	106.6
2 - 4	-318.1	437.9	1193.8	-318.1	437.9	1193.8
2 - 3	-177.3	509.0	1195.3	-177.3	509.0	1195.3
4 - 11	-16614.4	-15646.0	-14677.6	-16614.4	-15646.0	-14677.6
4 - 9	-12770.4	-11661.0	-10551.6	-12770.4	-11661.0	-10551.6
4 - 12	-6378.4	-5410.0	-4441.6	-6378.4	-5410.0	-4441.6
4 - 7	-2529.4	-1561.0	-592.6	-2529.4	-1561.0	-592.6
4 - 1	-1624.9	-927.1	-229.3	-1624.9	-927.1	-229.3
4 - 2	-1193.8	-437.9	318.1	-1193.8	-437.9	318.1
4 - 3	-705.3	71.1	847.6	-705.3	71.1	847.6
3 - 11	-16632.2	-15717.1	-14802.1	-16632.2	-15717.1	-14802.1
3 - 9	-12795.3	-11732.1	-10668.9	-12795.3	-11732.1	-10668.9
3 - 12	-6396.2	-5481.1	-4566.1	-6396.2	-5481.1	-4566.1
3 - 7	-2547.2	-1632.1	-717.1	-2547.2	-1632.1	-717.1
3 - 1	-1619.9	-998.3	-376.6	-1619.9	-998.3	-376.6
3 - 2	-1195.3	-509.0	177.3	-1195.3	-509.0	177.3
3 - 4	-847.6	-71.1	705.3	-847.6	-71.1	705.3

430

Method comparison

K

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

Class Level Information

Tukey's Studentized Range (HSD) Test for variable: K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 64 MSE= 622044
Critical Value of Studentized Range= 4.635

Number of observations in by group = 74

Comparisons significant at the 0.05 level are indicated by '***'.

Dependent Variable: K

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	57875652.5890	10.34	0.0001
Error	64	39810816.1553		
Corrected Total	73	97686468.7443		
R-Square			K Mean	
			C.V.	
			31.95182	2468.3964865

Source

Source	DF	Type I SS	F Value	Pr > F
METH	9	57875652.5890	10.34	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	9	57875652.5890	10.34	0.0001

Source

METH	9	57875652.5890	10.34	0.0001

5	-	11	-3920.1	-1809.3	301.4	***
5	-	6	-2988.4	-3.3	2981.7	***
5	-	7	-837.4	1273.3	3384.1	***
5	-	12	-513.5	1460.9	3435.3	***
5	-	9	-39.8	1744.1	3528.0	***
5	-	1	183.1	1746.2	3309.3	***
5	-	2	404.4	2106.1	3807.8	***
5	-	4	478.8	2366.7	4254.6	***
5	-	3	754.0	2537.9	4321.8	***
7	-	11	-5193.4	-3082.7	-971.9	***
7	-	6	-4261.7	-1276.7	1708.4	
7	-	5	-3384.1	-1273.3	837.4	
7	-	12	-1786.8	187.6	2162.0	
7	-	9	-1313.1	470.8	2254.7	
7	-	1	-1090.2	472.8	2035.9	
7	-	2	-869.0	832.8	2534.5	
7	-	4	-794.6	1093.3	2981.2	
7	-	3	-519.4	1264.5	3048.4	

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

METH Comparison		Simultaneous		Difference Between Means	Simultaneous Upper Confidence Limit
		Lower Confidence Limit	Upper Confidence Limit		
4	- 3	-1342.5	171.2	1684.9	***
3	- 11	-6131.1	-4347.2	-2563.3	***
3	- 6	-5304.8	-2541.2	222.4	***
3	- 5	-4321.8	-2537.9	-754.0	***
3	- 7	-3048.4	-1264.5	519.4	
3	- 12	-2697.3	-1077.0	543.4	
3	- 9	-2175.6	-793.8	588.0	
3	- 1	-1873.5	-791.7	290.1	
3	- 2	-1705.7	-431.8	842.2	
3	- 4	-1684.9	-171.2	1342.5	

METH Comparison		Simultaneous		Difference Between Means	Simultaneous Upper Confidence Limit
		Lower Confidence Limit	Upper Confidence Limit		
12	- 11	-5244.7	-3270.3	-1295.8	***
12	- 6	-4354.5	-1464.2	1426.0	
12	- 5	-3435.3	-1460.9	513.5	
12	- 7	-2162.0	-187.6	1786.8	
12	- 9	-1337.1	283.2	1903.5	
12	- 1	-1088.2	285.3	1658.7	
12	- 2	-884.2	645.2	2174.6	
12	- 4	-828.4	905.7	2639.9	
12	- 3	-543.4	1077.0	2697.3	
9	- 11	-5337.3	-3553.4	-1769.5	***
9	- 6	-4511.0	-1747.4	1016.2	
9	- 5	-3528.0	-1744.1	39.8	
9	- 7	-2254.7	-470.8	1313.1	
9	- 12	-1903.5	-283.2	1337.1	
9	- 1	-1079.7	2.1	1083.9	
9	- 2	-912.0	362.0	1636.0	
9	- 4	-891.1	622.6	2136.3	
9	- 3	-588.0	793.8	2175.6	
1	- 11	-5118.6	-3555.5	-1992.4	***
1	- 6	-4376.0	-1749.5	877.0	
1	- 5	-3309.3	-1746.2	-183.1	***
1	- 7	-2035.9	-472.8	1090.2	
1	- 12	-1658.7	-285.3	1088.2	
1	- 9	-1083.9	-2.1	1079.7	
1	- 2	-580.2	359.9	1300.1	
1	- 4	-625.4	620.5	1866.3	
1	- 3	-290.1	791.7	1873.5	
2	- 11	-5617.2	-3915.4	-2213.7	***
2	- 6	-4820.7	-2109.4	601.9	
2	- 5	-3807.8	-2106.1	-404.4	***
2	- 7	-2534.5	-832.8	869.0	
2	- 12	-2174.6	-645.2	884.2	
2	- 9	-1636.0	-362.0	912.0	
2	- 1	-1300.1	-359.9	580.2	
2	- 4	-1155.4	260.6	1676.5	
2	- 3	-842.2	431.8	1705.7	
4	- 11	-6063.9	-4176.0	-2288.1	***
4	- 6	-5201.9	-2370.0	461.9	
4	- 5	-4254.6	-2366.7	-478.8	***
4	- 7	-2981.2	-1093.3	794.6	
4	- 12	-2639.9	-905.7	828.4	
4	- 9	-2136.3	-622.6	891.1	
4	- 1	-1866.3	-620.5	625.4	
4	- 2	-1676.5	-260.6	1155.4	

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 11 12 2 3 4 7 9

Number of observations in by group = 44

Tukey's Studentized Range (HSD) Test for variable: K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 249101.6
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '****'.

Dependent Variable: K				Simultaneous				Simultaneous			
Source	DF	Sum of Squares	F Value	Pr > F	METH Comparison	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit			
Model	7	784327996.176	449.80	0.0001	11 - 9	9823.6	11288.7	12753.7	***		
Error	36	8967657.971			11 - 12	12359.3	13669.7	14980.0	***		
Corrected Total	43	793295654.147			11 - 7	13878.3	15188.7	16499.0	***		
					11 - 1	14849.8	15877.7	16905.7	***		
					11 - 4	15216.3	16388.3	17560.3	***		
					11 - 2	15308.1	16394.6	17481.1	***		
					11 - 3	16370.8	17478.2	18585.7	***		
R-Square		C.V.		K Mean							
0.988696		11.90312		4193.0252273	9 - 11	-12753.7	-11288.7	-9823.6	***		
					9 - 12	916.0	2381.0	3846.0	***		
					9 - 7	2435.0	3900.0	5365.0	***		
					9 - 1	3370.1	4589.1	5808.0	***		
					9 - 4	3756.9	5099.6	6442.3	***		
					9 - 2	3837.2	5105.9	6374.6	***		
					9 - 3	4902.8	6189.6	7476.3	***		
Source	DF	Type I SS	F Value	Pr > F	12 - 11	-14980.0	-13669.7	-12359.3	***		
					12 - 9	-3846.0	-2381.0	-916.0	***		
					12 - 7	208.6	1519.0	2829.4	***		
					12 - 1	1180.1	2208.1	3236.0	***		
					12 - 4	1546.6	2718.6	3890.6	***		
					12 - 2	1638.4	2724.9	3811.4	***		
					12 - 3	2701.1	3808.6	4916.0	***		
Source	DF	Type III SS	F Value	Pr > F	7 - 11	-16499.0	-15188.7	-13878.3	***		
					7 - 9	-5365.0	-3900.0	-2435.0	***		
					7 - 12	-2829.4	-1519.0	-208.6	***		
					7 - 1	-338.9	689.1	1717.0	***		
					7 - 4	27.6	1199.6	2371.6	***		
					7 - 2	119.4	1205.9	2292.4	***		
					7 - 3	1182.1	2289.6	3397.0	***		
Source	DF	Type III SS	F Value	Pr > F	1 - 11	-16905.7	-15877.7	-14849.8	***		
					1 - 9	-5808.0	-4589.1	-3370.1	***		
					1 - 12	-3236.0	-2208.1	-1180.1	***		
					1 - 7	-1717.0	-689.1	338.9	***		
					1 - 4	-334.0	510.5	1355.1	***		
					1 - 2	-204.3	516.8	1238.0	***		
					1 - 3	848.1	1600.5	2352.9	***		

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 11 12 2 3 4 7 9

Number of observations in by group = 44

Dependent Variable: K

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	64011695.5954	1113.47	0.0001
Error	36	295655.1953		
Corrected Total	43	64307350.7907		

R-Square C.V. K Mean

0.995402 12.43442 728.81295455

Source	DF	Type I SS	F Value	Pr > F
METH	7	64011695.5954	1113.47	0.0001

Source	DF	Type III SS	F Value	Pr > F
METH	7	64011695.5954	1113.47	0.0001

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Simultaneous Upper Confidence Limit	
	Lower Limit	Difference Between Means	Upper Limit	
4 - 11	-17560.3	-16388.3	-15216.3	***
4 - 9	-6442.3	-5099.6	-3756.9	***
4 - 12	-3890.6	-2718.6	-1546.6	***
4 - 7	-2371.6	-1199.6	-27.6	***
4 - 1	-1355.1	-510.5	334.0	
4 - 2	-908.6	6.3	921.2	
4 - 3	150.3	1090.0	2029.7	***
2 - 11	-17481.1	-16394.6	-15308.1	***
2 - 9	-6374.6	-5105.9	-3837.2	***
2 - 12	-3811.4	-2724.9	-1638.4	***
2 - 7	-2292.4	-1205.9	-119.4	***
2 - 1	-1238.0	-516.8	204.3	
2 - 2	-921.2	-6.3	908.6	
2 - 3	253.1	1083.7	1914.2	***
3 - 11	-18585.7	-17478.2	-16370.8	***
3 - 9	-7476.3	-6189.6	-4902.8	***
3 - 12	-4916.0	-3808.6	-2701.1	***
3 - 7	-3397.0	-2289.6	-1182.1	***
3 - 1	-2352.9	-1600.5	-848.1	***
3 - 2	-2029.7	-1090.0	-150.3	***
3 - 3	-1914.2	-1083.7	-253.1	***

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: K

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 8212.644

Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

Simultaneous

Difference

Between

Means

Simultaneous

Lower

Confidence

Limit

METH

Comparison

Simultaneous

Upper

Confidence

Limit

1	- 11	-4980.35	-4793.70	-4607.06	***
1	- 9	-884.37	-663.04	-441.70	***
1	- 12	-707.68	-521.04	-334.39	***
1	- 7	-349.68	-163.04	23.61	
1	- 4	-206.78	-53.44	99.91	
1	- 2	-81.59	49.36	180.30	
1	- 3	-80.84	55.77	192.38	
2	- 11	-5040.34	-4843.06	-4645.78	***
2	- 9	-942.76	-712.39	-482.02	***
2	- 12	-767.67	-570.39	-373.11	***
2	- 7	-409.67	-212.39	-15.11	***
2	- 4	-268.92	-102.79	63.33	
2	- 1	-180.30	-49.36	81.59	
2	- 3	-144.40	6.41	157.22	
3	- 11	-5050.55	-4849.47	-4648.39	***
3	- 9	-952.44	-718.80	-485.16	***
3	- 12	-777.89	-576.80	-375.72	***
3	- 7	-419.89	-218.80	-17.72	***
3	- 4	-279.83	-109.20	61.42	
3	- 1	-192.38	-55.77	80.84	
3	- 2	-157.22	-6.41	144.40	

Simultaneous

Difference

Between

Means

Simultaneous

Lower

Confidence

Limit

11	- 9	3864.66	4130.67	4396.68	***
11	- 12	4034.74	4272.67	4510.59	***
11	- 7	4392.74	4630.67	4868.59	***
11	- 4	4527.46	4740.27	4953.07	***
11	- 1	4607.06	4793.70	4980.35	***
11	- 2	4645.78	4843.06	5040.34	***
11	- 3	4648.39	4849.47	5050.55	***
9	- 11	-4396.68	-4130.67	-3864.66	***
9	- 12	-124.01	142.00	408.01	***
9	- 7	233.99	500.00	766.01	***
9	- 4	365.80	609.60	853.40	***
9	- 1	441.70	663.04	884.37	***
9	- 2	482.02	712.39	942.76	***
9	- 3	485.16	718.80	952.44	***
12	- 11	-4510.59	-4272.67	-4034.74	***
12	- 9	-408.01	-142.00	124.01	***
12	- 7	120.07	358.00	595.93	***
12	- 4	254.79	467.60	680.41	***
12	- 1	334.39	521.04	707.68	***
12	- 2	373.11	570.39	767.67	***
12	- 3	375.72	576.80	777.89	***
7	- 11	-4868.59	-4630.67	-4392.74	***
7	- 9	-766.01	-500.00	-233.99	***
7	- 12	-595.93	-358.00	-126.07	***
7	- 4	-103.21	109.60	322.41	
7	- 1	-23.61	163.04	349.68	
7	- 2	15.11	212.39	409.67	***
7	- 3	17.72	218.80	419.89	***
4	- 11	-4953.07	-4740.27	-4527.46	***
4	- 9	-853.40	-609.60	-365.80	***
4	- 12	-680.41	-467.60	-254.79	***
4	- 7	-322.41	-109.60	103.21	
4	- 1	-99.91	53.44	206.78	
4	- 2	-63.33	102.79	268.92	
4	- 3	-61.42	109.20	279.83	

23. Ca

units: mg/kg
mandatory for sample A2
reported by 21 laboratories

TABLE I : Ca - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N A2:N/R
2	A2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5	A2	1 not reported	N
6	A1 A2 B1 B2	2	N
7	A1 A2 B1 B2	3	N A2:N/R
8	A1 A2 B1 B2	3	N A2:N/R
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	N A2:R
11	A1 A2 B1 B2	1 not reported	N
12	A2	5	N
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	A1:3 A2:2 B1 B2:4	N
16	A1 A2 B1 B2	3	R A2:N/R
17	A2	3	N
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20	A2	1 not reported	N
21	A2	3	R
22	A2	3	R

(*) R = aqua regia

TABLE II : Ca - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	dry ashing
5	digestion, HNO3 under pressure
6	extractant HF
7	combustion, HClO4 + H2SO4
* 8	minéralisation
9	wet ashing
10	extractant 0.2N BaCl2 at pH 8.1
11	ignition, HF + HClO4
12	unspecified

* not applicable

TABLE IIIa : Ca - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	14000.00	14000.00	14000.00	0.00	0.00
2							
3	2	5	78373.23	79000.89	79674.63	526.65	0.67
4	3	4	63623.00	68764.00	76391.00	6252.73	9.09
5							
6	9	2	72650.00	72700.00	72750.00	70.71	0.10
7	11	3	79050.00	79390.00	79764.00	358.21	0.45
8	12	3	100000.00	100000.00	100000.00	0.00	0.00
9	2	3	73400.00	74000.00	74500.00	556.78	0.75
10							
11							
12							
13							
14	4	5	73995.00	76242.60	77783.00	1458.39	1.91
15	2	4	61044.00	65552.50	68816.00	3511.92	5.36
16							
17							
18	3	3	75590.40	76098.40	76403.20	442.86	0.58
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	5700.00	6400.00	7800.00	1212.44	18.94
2	2	3	10488.00	10856.00	11592.00	637.40	5.87
3	2	5	14995.93	15475.69	16785.46	655.71	4.24
4	3	4	14489.00	14662.50	14882.00	201.40	1.37
5	12	1	11949.00	11949.00	11949.00	0.00	0.00
6	9	2	14000.00	14200.00	14400.00	282.84	1.99
7	11	3	14285.00	15108.00	15624.00	720.36	4.77
8	12	3	5040.00	5040.00	5040.00	0.00	0.00
9	2	3	14800.00	15066.67	15300.00	251.66	1.67
10							
11							
12	10	5	17978.00	18049.20	18200.00	89.06	0.49
13							
14	4	5	12847.00	13410.80	14073.00	463.72	3.46
14	A3	4	13858.00	14026.40	14221.00	133.02	0.95
15	2	2	14376.00	14728.00	15080.00	497.80	3.38
16	5	3	13950.00	14030.00	14110.00	80.00	0.57
17							
18	3	3	12717.00	13079.93	13370.10	332.58	2.54
19							
20	8	1		13775.00			
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	500.00	500.00	500.00	0.00	0.00
2							
3	2	5	690.58	726.85	773.94	28.46	3.92
4	3	4	375.00	388.75	400.00	11.41	2.94
5							
6	9	2	1550.00	1600.00	1850.00	70.71	4.42
7	11	3	2346.00	2414.00	2448.00	58.89	2.44
8	12	3	2040.00	2074.80	2092.40	30.14	1.45
9	2	3	580.00	650.00	720.00	70.00	10.77
10							
11							
12							
13							
14	4	5	326.00	346.80	370.00	15.81	4.56
15	2	4	375.40	422.13	477.50	51.34	12.16
16							
17							
18	3	3	507.50	512.58	516.64	4.65	0.91
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	100.00	100.00	100.00	0.00	0.00
2							
3	2	5	385.97	432.86	514.63	46.89	10.83
4	3	4	402.00	418.00	429.00	11.46	2.74
5							
6	9	2	600.00	700.00	800.00	141.42	20.20
7	11	3	714.00	781.33	816.00	58.32	7.46
8	12	3	1040.00	1373.33	2040.00	577.35	42.04
9	2	3	360.00	406.67	500.00	80.83	19.88
10							
11							
12							
13							
14	4	5	388.00	401.80	438.00	20.63	5.14
15	2	4	281.40	302.83	315.10	15.43	5.10
16							
17							
18	3	3	368.37	381.86	394.68	13.17	3.45
19							
20							
21							
22							

TABLE IIIb : Ca - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	70710.00	71776.67	72710.00	1006.65	1.40
11	3	73650.00	74466.67	75400.00	880.81	1.18
12						
13						
14						
15						
16	3	77010.00	77403.33	77880.00	440.95	0.57
17						
18	3	68173.60	66817.07	69291.20	577.72	0.84
19	3		54898.00			
20						
21						
22						

sample A2 - reference method

labcode	method	minimum	mean	maximum	std	cv
1	3	2800.00	3133.33	3700.00	493.29	15.74
2						
3						
4						
5						
6						
7	3	13726.00	14582.00	15066.00	743.43	5.10
8	3	6790.00	6794.67	6797.00	4.04	0.06
9						
10	3	16905.00	17305.00	17705.00	400.00	2.31
11	3	12300.00	12466.67	12600.00	152.75	1.23
12						
13						
14						
15						
16	3	12280.00	12443.33	12770.00	282.90	2.27
17	3	11600.00	12500.00	13200.00	818.54	6.55
18	3	12174.40	12246.87	12391.80	125.52	1.03
19	3		10012.00			
20						
21	3	4360.00	4360.00	4360.00	0.00	0.00
22	3	20021.00	20095.67	20213.00	102.87	0.51

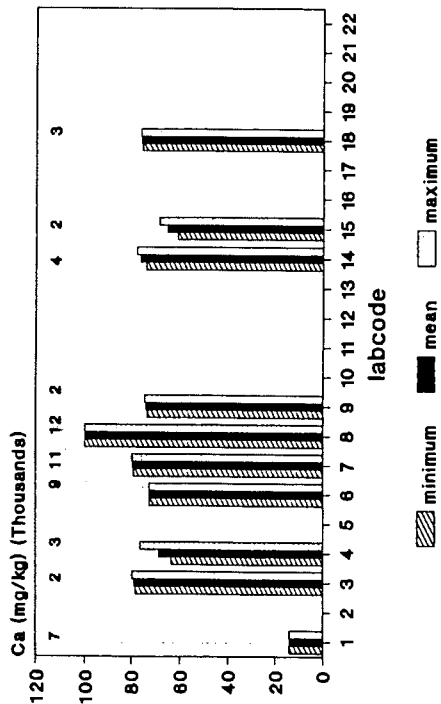
sample B1 - reference method

labcode	method	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	459.00	538.33	641.00	93.22	17.32
11	3	590.00	630.00	670.00	40.00	6.35
12						
13						
14						
15						
16	3	710.00	722.67	735.00	12.50	1.73
17						
18	3	951.06	988.61	1018.05	34.23	3.46
19	3		675.00			
20						
21						
22						

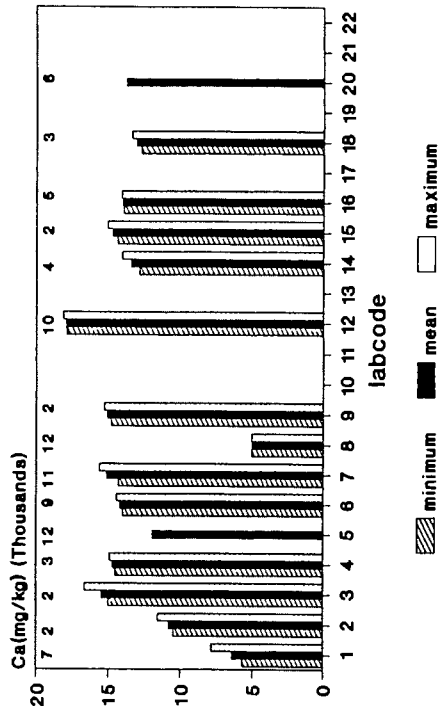
sample B2 - reference method

labcode	method	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	734.00	856.67	985.00	125.60	14.88
11	3	430.00	453.33	480.00	25.17	5.55
12						
13						
14						
15						
16	3	391.00	407.00	418.00	14.18	3.48
17						
18	3	586.96	626.77	661.85	37.67	6.01
19	3		496.00			
20						
21						
22						

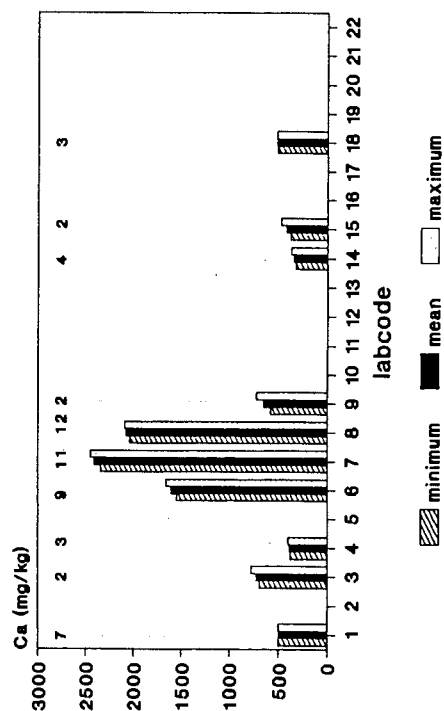
Ca sample A1
national methods



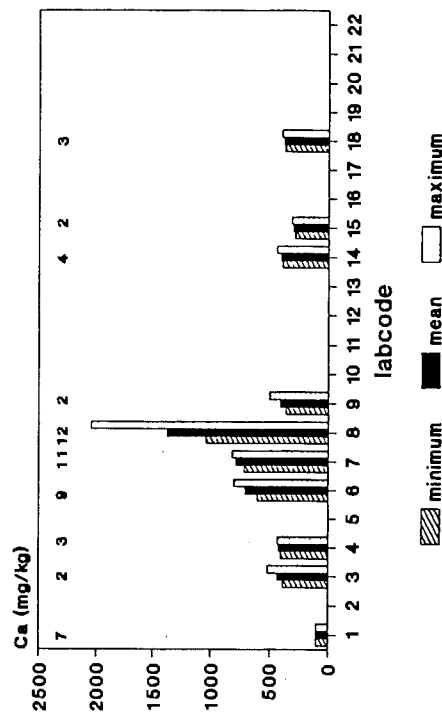
Ca sample A2
national methods



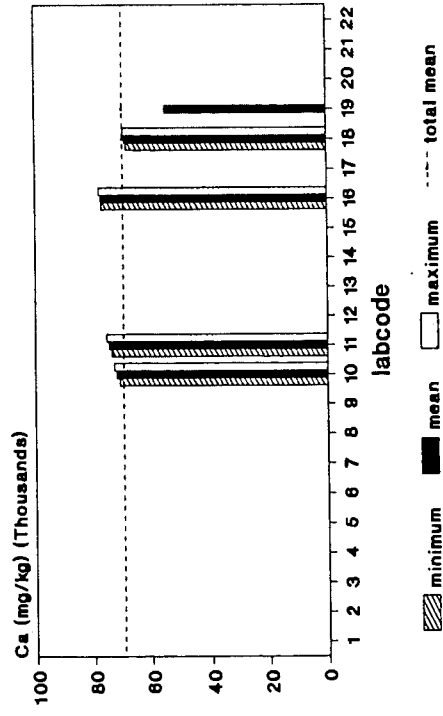
Ca sample B1
national methods



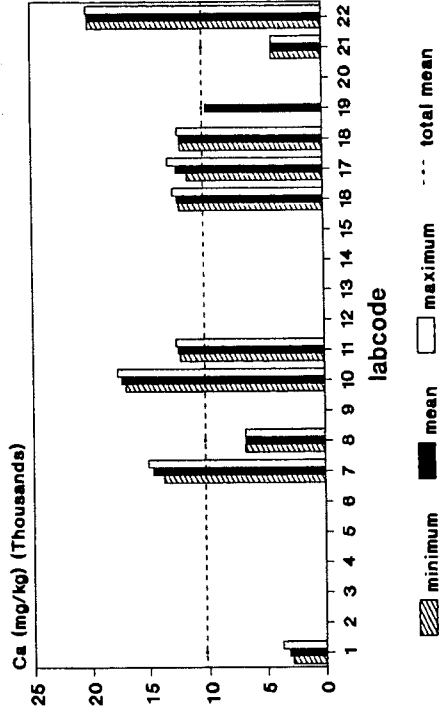
Ca sample B2
national methods



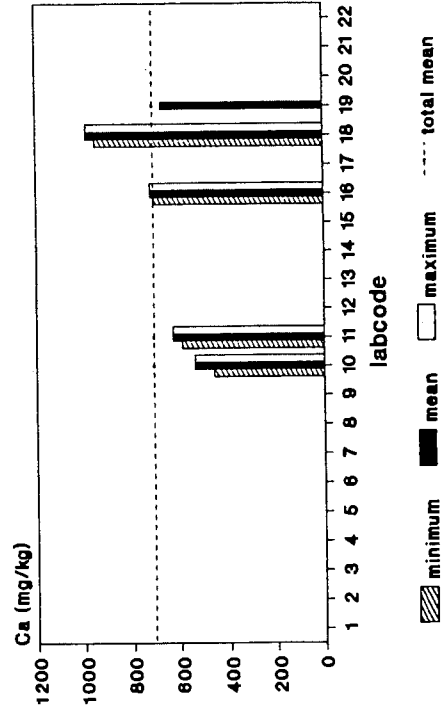
Ca sample A1
reference method



Ca sample A2
reference method



Ca sample B1
reference method



Ca sample B2
reference method

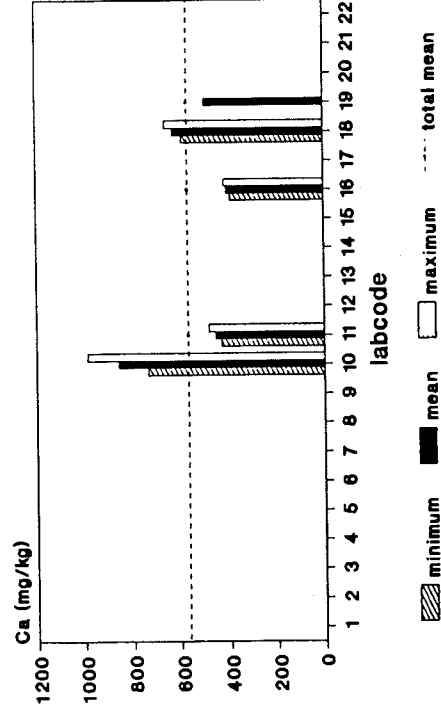


TABLE V : Ca Method comparison

SAMPLE=Al

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 11 12 2 3 4 7 9

Number of observations in by group = 44

Dependent Variable: Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	12631981795.4	72.54	0.0001
Error	36	895575911.4		
Corrected Total	43	13527557706.8		

R-Square C.V. Ca Mean

0.933796 7.066467 70582.597500

Source	DF	Type I SS	F Value	Pr > F
METH	7	12631981795.4	72.54	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	7	12631981795.4	72.54	0.0001

TABLE IV : Ca - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	71714.55	6003.04	0.82	0.01
A1	2	8	70401.36	5909.67	0.97	0.87
A1	3	7	71907.31	5914.66	0.74	0.01
A1	4	5	76242.60	1458.39	0.95	0.73
A1	7	3	14000.00	0.00		
A1	9	2	72700.00	70.71	1.00	1.00
A1	11	3	79390.00	358.21	0.99	0.84
A1	12	3	100000.00	0.00		
A2	1	31	11541.76	5152.43	0.92	0.03
A2	2	9	13633.30	2129.61	0.77	0.01
A2	3	7	13984.26	879.05	0.86	0.16
A2	4	5	13410.80	463.72	0.98	0.92
A2	5	3	14030.00	80.00	1.00	0.99
A2	6	1	13775.00			
A2	7	3	6400.00	1212.44	0.75	0.00
A2	9	7	15949.43	1883.17	0.65	0.00
A2	11	3	15108.00	720.36	0.86	0.28
A2	12	4	6767.25	3454.50	0.63	0.00
A3	4	5	14024.40	133.02	0.96	0.81
B1	1	13	716.45	174.38	0.91	0.18
B1	2	8	545.64	143.51	0.90	0.29
B1	3	7	441.82	66.73	0.76	0.02
B1	4	5	346.60	15.81	0.96	0.79
B1	7	3	500.00	0.00		
B1	9	2	1600.00	70.71	1.00	1.00
B1	11	3	2414.00	58.89	0.75	0.00
B1	12	3	2074.80	30.14	0.76	0.01
B2	1	13	579.02	186.46	0.88	0.07
B2	2	8	358.02	74.32	0.88	0.19
B2	3	7	402.51	22.28	0.95	0.71
B2	4	5	401.80	20.63	0.73	0.02
B2	7	3	100.00	0.00		
B2	9	2	700.00	141.42	1.00	1.00
B2	11	3	781.33	58.32	0.77	0.03
B2	12	3	1373.33	577.35	0.75	0.00

Remark:

Labcode 3 and labcode 19 reported mean values for resp. 5 and 3 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for methods 2 and 1 are different from the real number of replicates.

SAMPLE=AL

SAMPLE=AL

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 24877109
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
12 - 11	7515	20610	33705	***		
12 - 4	12045	23757	35470	***		
12 - 9	12660	27300	41940	***		
12 - 3	17026	28093	39160	***		
12 - 1	18013	28285	38558	***		
12 - 2	18741	29599	40456	***		
12 - 7	72905	86000	99095	***		
11 - 12	-33705	-20610	-7515	***		
11 - 4	-8565	3147	14860			
11 - 9	-7950	6690	21330			
11 - 3	-3584	7483	18550			
11 - 1	-2597	7675	17948			
11 - 2	-1869	8989	19846			
11 - 7	52295	65390	78485	***		
4 - 12	-35470	-23757	-12045	***		
4 - 11	-14860	-3147	8565			
4 - 9	-9876	3543	16961			
4 - 3	-5056	4335	13726			
4 - 1	-3912	4528	12968			
4 - 2	-3302	5841	14984	***		
4 - 7	50530	62243	73955	***		
9 - 12	-41940	-27300	-12660	***		
9 - 11	-21330	-6690	7950			
9 - 4	-16961	-3543	9876			
9 - 3	-12066	793	13652			
9 - 1	-11196	985	13167			
9 - 2	-10380	2299	14978	***		
9 - 7	44060	58700	73340	***		
3 - 12	-39160	-28093	-17026	***		
3 - 11	-18550	-7483	3584			
3 - 4	-13726	-4335	5056			
3 - 9	-13652	-793	12066			
3 - 1	-7326	193	7711			
3 - 2	-6794	1506	9806	***		
3 - 7	46840	57907	68974	***		

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 12	-38558	-28285	-18013	***		
1 - 11	-17948	-7675	2597			
1 - 4	-12968	-4528	3912			
1 - 9	-13167	-985	11196			
1 - 3	-7711	-193	7326			
1 - 2	-5894	1313	8520	***		
1 - 7	47442	57715	67987	***		
2 - 12	-40456	-29599	-18741	***		
2 - 11	-19846	-8989	1869			
2 - 4	-14984	-5841	3302			
2 - 9	-14978	-2299	10380			
2 - 3	-9806	-1506	6794			
2 - 1	-8520	-1313	5894	***		
2 - 7	45544	56401	67259	***		
7 - 12	-99095	-86000	-72905	***		
7 - 11	-78485	-65390	-52295	***		
7 - 4	-73955	-62243	-50530	***		
7 - 9	-73340	-58700	-44060	***		
7 - 3	-68974	-57907	-46840	***		
7 - 1	-67987	-57715	-47442	***		
7 - 2	-67259	-56401	-45544	***		

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 63 MSE= 14274206
Critical Value of Studentized Range= 4.638

Comparisons significant at the 0.05 level are indicated by '****'.

Simultaneous

METH Comparison	Simultaneous		Difference Between Means	Upper Limit
	Lower Limit	Confidence Limit		
9 - 11	-6708.9	1841.4	10391.7	10391.7
9 - 5	-5630.9	2919.4	11469.7	11469.7
9 - 3	-3657.9	2965.2	9588.2	9588.2
9 - 6	-10071.6	3174.4	16420.5	16420.5
9 - 2	-2928.1	3316.1	9560.4	9560.4
9 - 4	-3716.5	3538.6	10793.8	10793.8
9 - 1	222.6	5407.7	10592.7	10592.7
9 - 12	2416.0	10182.2	17948.4	17948.4
9 - 7	1999.1	10549.4	19099.7	19099.7

Simultaneous

METH Comparison	Simultaneous		Difference Between Means	Upper Limit
	Lower Limit	Confidence Limit		
11 - 9	-10391.7	-1841.4	6708.9	6708.9
11 - 5	-9038.8	1078.0	11194.8	11194.8
11 - 3	-7426.6	1123.7	9674.0	9674.0
11 - 6	-12974.4	1333.0	15640.4	15640.4
11 - 2	-6785.7	1474.7	9735.1	9735.1
11 - 4	-7351.6	1697.2	10746.0	10746.0
11 - 1	-3925.6	3566.2	11058.1	11058.1
11 - 12	-1122.7	8340.7	17804.2	17804.2
11 - 7	-1408.8	8708.0	18824.8	18824.8

Simultaneous

METH Comparison	Simultaneous		Difference Between Means	Upper Limit
	Lower Limit	Confidence Limit		
5 - 9	-11469.7	-2919.4	5630.9	5630.9
5 - 11	-11194.8	-1078.0	9038.8	9038.8
5 - 3	-8504.6	45.7	8596.0	8596.0
5 - 6	-14052.4	255.0	14562.4	14562.4
5 - 2	-7863.7	396.7	8657.1	8657.1
5 - 4	-8429.6	619.2	9668.0	9668.0
5 - 1	-5003.6	2488.2	9980.1	9980.1
5 - 12	-2200.7	7262.8	16726.2	16726.2
5 - 7	-2486.8	7630.0	17746.8	17746.8

Simultaneous

METH Comparison	Simultaneous		Difference Between Means	Upper Limit
	Lower Limit	Confidence Limit		
3 - 9	-9588.2	-2965.2	3657.9	3657.9
3 - 11	-9674.0	-1123.7	7426.6	7426.6
3 - 5	-8596.0	-45.7	8504.6	8504.6
3 - 6	-13036.8	209.3	13455.3	13455.3
3 - 2	-5893.3	351.0	6595.2	6595.2
3 - 4	-6681.7	573.5	7828.6	7828.6
3 - 1	-2742.6	2442.5	7627.5	7627.5
3 - 12	-549.2	7217.0	14983.2	14983.2
3 - 7	-966.0	7584.3	16134.6	16134.6

SAMPLE=A2

General Linear Models Procedure

Class Level Information

Class Levels Values

METH 10 1 11 12 2 3 4 5 6 7 9

Number of observations in by group = 73

Dependent Variable: Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	470303419.362	3.66	0.0010
Error	63	899274994.127		
Corrected Total	72	1369578413.489		
R-Square		C.V.	Ca Mean	
0.343393		30.25674	12486.877945	

Source	DF	Type I SS	F Value	Pr > F
METH	9	470303419.362	3.66	0.0010
Source	DF	Type III SS	F Value	Pr > F
METH	9	470303419.362	3.66	0.0010

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
12 - 7	-9096.2	367.3	9830.7
7 - 9	-19099.7	-10549.4	-1999.1 ***
7 - 11	-18824.8	-8708.0	1408.8
7 - 5	-17746.8	-7630.0	2486.8
7 - 3	-16134.6	-7584.3	966.0
7 - 6	-21682.4	-7375.0	6932.4
7 - 2	-15493.7	-7233.3	1027.1
7 - 4	-16059.6	-7010.8	2038.0
7 - 1	-12633.6	-5141.8	2350.1
7 - 12	-9830.7	-367.3	9096.2

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
6 - 9	-16420.5	-3174.4	10071.6
6 - 11	-15640.4	-1333.0	12974.4
6 - 5	-14562.4	-255.0	14052.4
6 - 3	-13455.3	-209.3	13036.8
6 - 2	-12919.1	141.7	13202.5
6 - 4	-13209.0	364.2	13937.4
6 - 1	-10355.6	2233.2	14822.1
6 - 12	-6845.3	7007.8	20860.8
6 - 7	-6932.4	7375.0	21682.4
2 - 9	-9560.4	-3316.1	2928.1
2 - 11	-9735.1	-1474.7	6785.7
2 - 5	-8657.1	-396.7	7863.7
2 - 3	-6595.2	-351.0	5893.3
2 - 6	-13202.5	-141.7	12919.1
2 - 4	-6688.6	222.5	7133.6
2 - 1	-2600.0	2091.5	6783.1
2 - 12	-579.7	6866.0	14311.8
2 - 7	-1027.1	7233.3	15493.7
4 - 9	-10793.8	-3538.6	3716.5
4 - 11	-10746.0	-1697.2	7351.6
4 - 5	-9668.0	-619.2	8429.6
4 - 3	-7828.6	-573.5	6681.7
4 - 6	-13937.4	-364.2	13209.0
4 - 2	-7133.6	-222.5	6688.6
4 - 1	-4102.4	1869.0	7840.4
4 - 12	-1668.3	6643.5	14955.4
4 - 7	-2038.0	7010.8	16059.6
1 - 9	-10592.7	-5407.7	-222.6 ***
1 - 11	-11058.1	-3566.2	3925.6
1 - 5	-9980.1	-2488.2	5003.6
1 - 3	-7627.5	-2442.5	2742.6
1 - 6	-14822.1	-2233.2	10355.6
1 - 2	-6783.1	-2091.5	2600.0
1 - 4	-7840.4	-1869.0	4102.4
1 - 12	-1808.3	4774.5	11357.4
1 - 7	-2350.1	5141.8	12633.6
12 - 9	-17948.4	-10182.2	-2416.0 ***
12 - 11	-17804.2	-8340.7	1122.7
12 - 5	-16726.2	-7262.8	2200.7
12 - 3	-14983.2	-7217.0	549.2
12 - 6	-20860.8	-7007.8	6845.3
12 - 2	-14311.8	-6866.0	579.7
12 - 4	-14955.4	-6643.5	1668.3
12 - 1	-11357.4	-4774.5	1808.3

Ca Method comparison

Ca Method comparison

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Class	Levels	Values
METH	8	1 11 12 2 3 4 7 9

Tukey's Studentized Range (HSD) Test for variable: Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 15292.35
Critical Value of Studentized Range= 4.547

Number of observations in by group = 45

Comparisons significant at the 0.05 level are indicated by '***'.

NOTE: Due to missing values, only 44 observations can be used in this analysis.

Dependent Variable: Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	16725388.5607	156.24	0.0001
Error	36	550524.6100		
Corrected Total	43	17275913.1707		

R-Square	C.V.	Ca Mean
0.968133	14.83767	833.43454545

Source	DF	Type I SS	F Value	Pr > F
METH	7	16725388.5607	156.24	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	7	16725388.5607	156.24	0.0001

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Limit	Confidence Limit	Between Means	Upper Confidence Limit	Upper Limit
11 - 12	14.53		339.20		663.87 ***
11 - 9	451.01		814.00		1176.99 ***
11 - 1	1442.86		1697.55		1952.24 ***
11 - 2	1599.16		1868.36		2137.56 ***
11 - 7	1589.33		1914.00		2238.67 ***
11 - 3	1697.79		1972.18		2246.57 ***
11 - 4	1777.01		2067.40		2357.79 ***
12 - 11	-663.87		-339.20		-14.53 ***
12 - 9	111.81		474.80		837.79 ***
12 - 1	1103.66		1358.35		1613.04 ***
12 - 2	1259.96		1529.16		1798.36 ***
12 - 7	1250.13		1574.80		1899.47 ***
12 - 3	1358.59		1632.98		1907.37 ***
12 - 4	1437.81		1728.20		2018.59 ***
9 - 11	-1176.99		-814.00		-451.01 ***
9 - 12	-837.79		-474.80		-111.81 ***
9 - 1	581.53		883.55		1185.58 ***
9 - 2	740.00		1054.36		1368.71 ***
9 - 7	737.01		1100.00		1462.99 ***
9 - 3	839.37		1158.18		1477.00 ***
9 - 4	920.72		1253.40		1586.08 ***
1 - 11	-1952.24		-1697.55		-1442.86 ***
1 - 12	-1613.04		-1358.35		-1103.66 ***
1 - 9	-1185.58		-883.55		-581.53 ***
1 - 2	-7.87		170.81		349.49
1 - 7	-38.24		216.45		471.14
1 - 3	88.22		274.63		461.04 ***
1 - 4	160.60		369.85		579.10 ***
2 - 11	-2137.56		-1868.36		-1599.16 ***
2 - 12	-1798.36		-1529.16		-1259.96 ***
2 - 9	-1368.71		-1054.36		-740.00 ***
2 - 1	-349.49		-170.81		7.87
2 - 7	-223.56		45.64		314.84
2 - 3	-101.97		103.83		309.62
2 - 4	-27.64		199.04		425.73

SAMPLE=B2

SAMPLE=B1

General Linear Models Procedure
Class Level Information

General Linear Models Procedure

Class	Levels	Values
METH	8	1 11 12 2 3 4 7 9

Simultaneous

Lower Confidence Limit	Difference Between Means	Upper Confidence Limit

Number of observations in by group = 45

NOTE: Due to missing values, only 44 observations can be used in this analysis.

Dependent Variable: Ca

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	3399357.53334	15.15	0.0001
Error	36	1154032.64675		
Corrected Total	43	4553390.18010		

R-Square C.V. Ca Mean

0.746555 33.69209 531.41022727

Source	DF	Type I SS	F Value	Pr > F
METH	7	3399357.53334	15.15	0.0001
Source	DF	Type III SS	F Value <td>Pr > F</td>	Pr > F
METH	7	3399357.53334	15.15	0.0001

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Ca

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 32056.46
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

General Linear Models Procedure			
METH Comparison	Simultaneous		Simultaneous
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 12	-1391.97	-971.53	-551.09 ***
4 - 11	-799.97	-379.53	40.91
4 - 9	-779.87	-298.20	183.47
4 - 1	-480.18	-177.22	125.74
4 - 3	-337.81	-0.71	336.39
4 - 2	-284.43	43.78	371.99
4 - 7	-118.64	301.80	722.24
2 - 12	-1405.07	-1015.31	-625.56 ***
2 - 11	-813.07	-423.31	-33.56 ***
2 - 9	-797.12	-341.98	113.16
2 - 1	-479.70	-221.00	37.70
2 - 3	-342.45	-44.49	253.47
2 - 4	-371.99	-43.78	284.43
2 - 7	-131.74	258.02	647.78
7 - 12	-1743.40	-1273.33	-803.27 ***
7 - 11	-1151.40	-681.33	-211.27 ***
7 - 9	-1125.55	-600.00	-74.45 ***
7 - 1	-847.77	-479.02	-110.27 ***
7 - 3	-699.79	-302.51	94.76
7 - 4	-722.24	-301.80	118.64
7 - 2	-647.78	-258.02	131.74

General Linear Models Procedure			
METH Comparison	Simultaneous		Simultaneous
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
12 - 11	121.93	592.00	1062.07 ***
12 - 9	147.78	673.33	1198.88 ***
12 - 1	425.56	794.31	1163.06 ***
12 - 3	573.54	970.82	1368.10 ***
12 - 4	551.09	971.53	1391.97 ***
12 - 2	625.56	1015.31	1405.07 ***
12 - 7	803.27	1273.33	1743.40 ***
11 - 12	-1062.07	-592.00	-121.93 ***
11 - 9	-444.22	81.33	606.88 ***
11 - 1	-166.44	202.31	571.06 ***
11 - 3	-18.46	378.82	776.10 ***
11 - 4	-40.91	379.53	799.97 ***
11 - 2	33.56	423.31	813.07 ***
11 - 7	211.27	681.33	1151.40 ***
9 - 12	-1198.88	-673.33	-147.78 ***
9 - 11	-606.88	-81.33	444.22
9 - 1	-316.31	120.98	558.26
9 - 3	-164.11	297.49	759.08
9 - 4	-183.47	298.20	779.87
9 - 2	-113.16	341.98	797.12
9 - 7	74.45	600.00	1125.55 ***
1 - 12	-1163.06	-794.31	-425.56 ***
1 - 11	-571.06	-202.31	166.44
1 - 9	-558.26	-120.98	316.31
1 - 3	-93.39	176.51	446.41
1 - 4	-125.74	177.22	480.18
1 - 2	-37.70	221.00	479.70
1 - 7	110.27	479.02	847.77 ***
3 - 12	-1368.10	-970.82	-573.54 ***
3 - 11	-776.10	-378.82	18.46
3 - 9	-759.08	-297.49	164.11
3 - 1	-446.41	-176.51	93.39
3 - 4	-336.39	0.71	337.81
3 - 2	-253.47	44.49	342.45
3 - 7	-94.76	302.51	699.79

24. Mg

units: mg/kg
mandatoy for sample A2
reported by 21 laboratories

TABLE I : Mg - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N A2:N/R
2	A2	3	N
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5	A2	1	N
6	A1 A2 B1 B2	2	N
7	A1 A2 B1 B2	3	N A2:N/R
8	A1 A2 B1 B2	3	N A2:N/R
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11	A1 A2 A2 B1	3	N
12	A2	5	N
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	4 A2:2	N
16	A1 A2 B1 B2	3	R A2:N/R
17	A2	3	R
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20	A2	1 not reported	N
21	A2	3	R
22	A2	3	R

(*) R = aqua regia

TABLE II : Mg - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	dry ashing
5	digestion, HNO3 under pressure
6	extractant HF
7	combustion, HClO4 + H2SO4
8	minéralisation
9	wet ashing
10	extractant 0.2N BaCl2 at pH 8.1
11	ignition, HF + HClO4
12	unspecified

TABLE IIIa : Mg - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	7	3	23120.00	23348.67	23460.00	196.30	0.84
2							
3	2	5	31381.89	31508.03	31722.64	121.28	0.38
4	3	4	23569.00	25917.00	28797.00	2396.95	9.25
5							
6	9	2	29700.00	29900.00	30100.00	282.84	0.95
7	10	3	31987.00	32123.33	32212.00	119.84	0.37
8	8	3	25000.00	25000.00	25000.00	0.00	0.00
9	2	3	28600.00	29866.67	30700.00	1115.05	3.73
10							
11							
12							
13							
14	4	5	24788.00	26271.20	27207.00	893.14	3.40
15	2	4	28734.00	30281.25	31428.00	1363.93	4.50
16							
17							
18	3	3	24892.00	25081.33	25298.40	211.50	0.84
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	2530.00	2610.00	2710.00	91.85	3.51
2	2	3	2760.00	2833.87	2981.00	127.59	4.50
3	2	5	2865.32	3099.62	3296.97	175.00	5.65
4	3	4	2220.00	2292.25	2394.00	69.06	3.01
5	10	1	1849.00				
6	9	2	2000.00	2200.00	2400.00	282.84	12.86
7	10	3	3150.00	3516.33	3984.00	426.13	12.12
8	8	3	2240.00	2240.00	2240.00	0.00	0.00
9	2	3	2530.00	2540.00	2550.00	10.00	0.39
10							
11							
12	10	5	2447.20	2404.00	2493.00	38.21	1.52
13							
14	4	5	2303.00	2441.80	2557.00	125.80	5.15
14	A3	4	2337.00	2428.60	2495.00	76.20	3.14
15	2	2	2539.00	2552.50	2566.00	19.09	0.75
16	5	3	2910.00	3000.00	3180.00	155.89	5.20
17							
18	3	3	1871.81	1936.31	1987.04	58.64	3.04
19							
20	6	1		2250.00			
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	2860.00	2860.00	2860.00	0.00	0.00
2							
3	2	5	3400.76	3471.00	3567.49	61.25	1.76
4	3	4	2193.00	2242.00	2334.00	62.85	2.80
5							
6	9	2	4300.00	4300.00	4300.00	0.00	0.00
7	10	3	4498.00	4515.00	4549.00	29.45	0.65
8	8	3	5100.00	5268.67	5406.00	155.39	2.95
9	2	3	3180.00	3183.33	3230.00	40.42	1.27
10							
11							
12							
13							
14	4	5	2310.00	2466.80	2565.00	99.95	4.05
15	2	4	2530.00	3052.25	3775.00	529.82	17.36
16							
17							
18	3	3	2366.98	2390.33	2416.72	25.01	1.05
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	330.00	330.00	330.00	0.00	0.00
2							
3	2	5	268.14	280.48	313.53	17.50	6.24
4	3	4	194.00	201.75	211.00	7.04	3.49
5							
6	9	2	500.00	500.00	500.00	0.00	0.00
7	10	3	418.00	418.00	418.00	0.00	0.00
8	8	3	371.24	381.42	397.02	13.72	3.60
9	2	3	240.00	240.00	240.00	0.00	0.00
10							
11							
12							
13							
14	4	5	205.00	215.20	221.00	6.10	2.83
15	2	4	179.40	185.55	195.80	7.10	3.83
16							
17							
18	3	3	176.09	188.91	196.33	11.15	5.90
19							
20							
21							
22							

TABLE IIIb : Mg - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	31030.00	32130.00	33550.00	1290.12	4.02
11	3	5830.00	5966.67	6160.00	172.14	2.89
12						
13						
14						
15						
16	3	29430.00	29596.67	29750.00	160.42	0.54
17						
18	3	25603.20	25738.67	26009.60	234.64	0.91
19	3		27266.00			
20						
21						
22						

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1	3	2470.00	2503.33	2570.00	57.74	2.31
2						
3						
4						
5						
6						
7	3	2723.00	2968.33	3180.00	230.35	7.76
8	3	2251.00	3368.00	4816.00	1314.15	39.02
9						
10	3	2830.00	2970.00	3190.00	182.87	6.49
11	3	2240.00	2310.00	2360.00	62.45	2.70
12						
13						
14						
15						
16	3	2180.00	2586.67	2800.00	352.33	13.62
17	3	2780.00	2853.33	2970.00	102.14	3.58
18	3	2505.54	2523.65	2532.71	15.69	0.62
19	3		267200.00			
20						
21	3	2350.00	2350.00	2350.00	0.00	0.00
22	3	6001.00	6062.00	6121.00	60.03	0.99

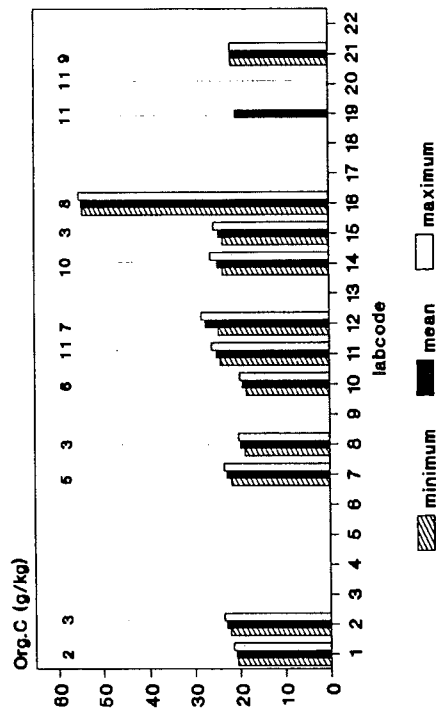
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	3420.00	3723.33	4075.00	330.16	8.87
11	3	3260.00	3346.67	3430.00	85.05	2.54
12						
13						
14						
15						
16	3	3270.00	3366.67	3420.00	83.87	2.49
17						
18	3	3018.61	3150.90	3347.47	173.60	5.51
19	3		2526.00			
20						
21						
22						

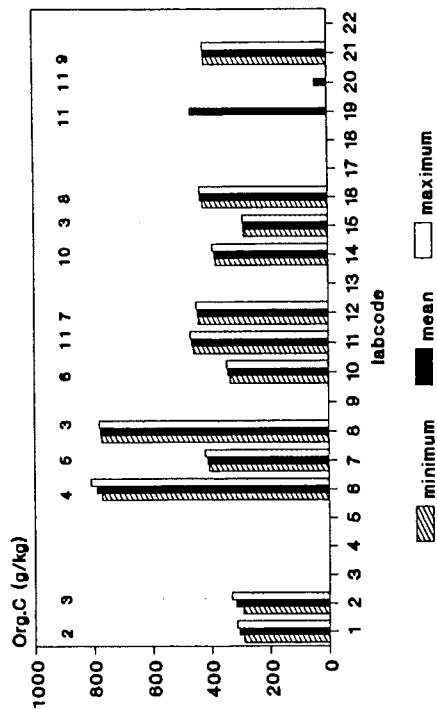
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	230.00	266.67	335.00	59.23	22.21
11	3	220.00	223.33	230.00	5.77	2.59
12						
13						
14						
15						
16	3	256.00	261.67	265.00	4.93	1.89
17						
18	3	220.62	223.65	225.68	2.68	1.20
19	3		250.00			
20						
21						
22						

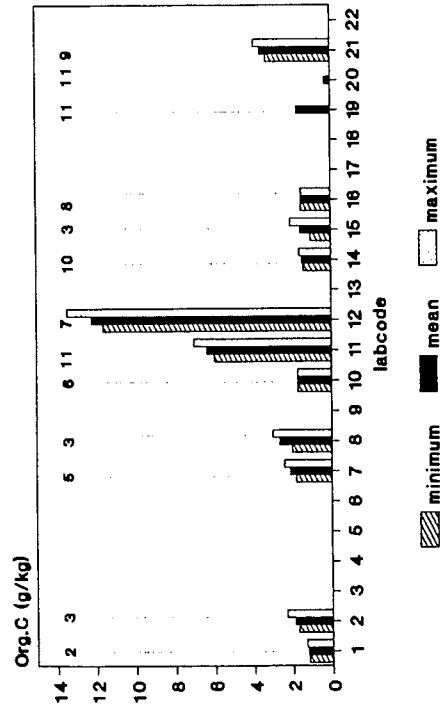
Org.C sample A1
national methods



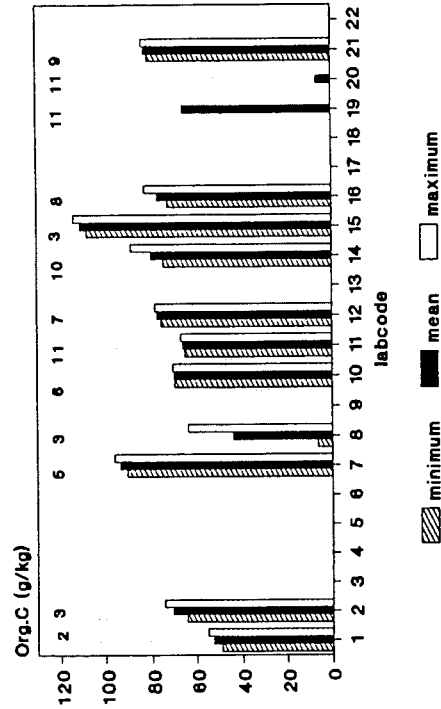
Org.C sample A2
national methods



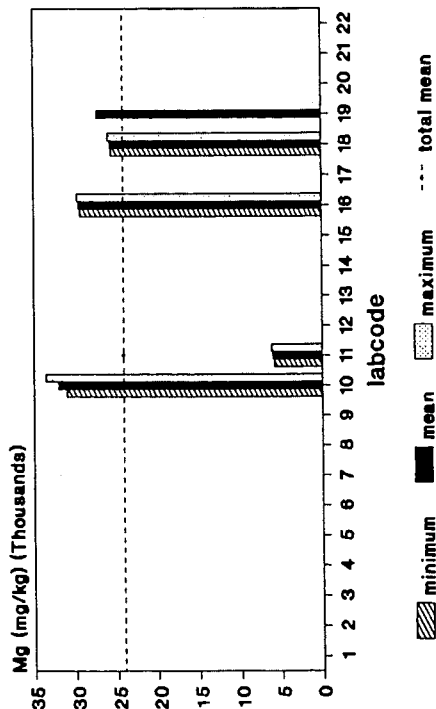
Org.C sample B1
national methods



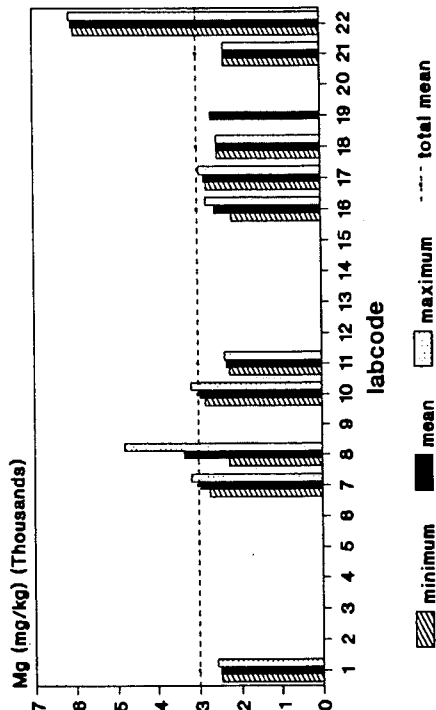
Org.C sample B2
national methods



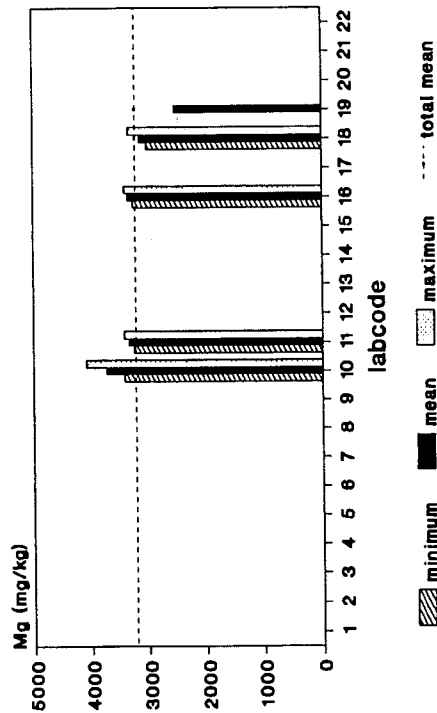
Mg sample A1
reference method



Mg sample A2
reference method



Mg sample B1
reference method



Mg sample B2
reference method

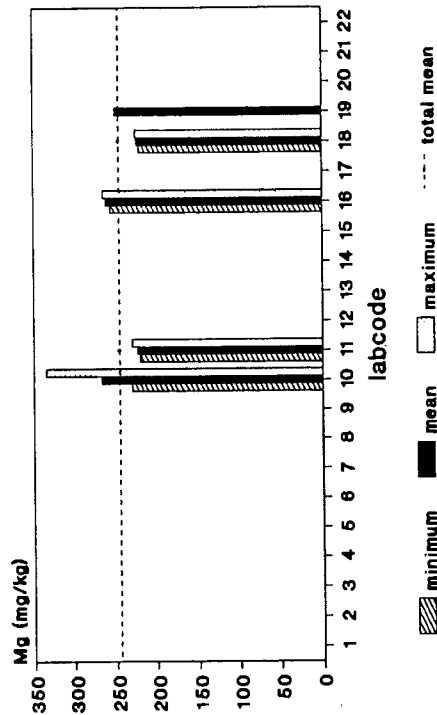


TABLE V : Mg Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 10 2 3 4 7 8 9

Number of observations in by group = 44

Dependent Variable: Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	379830394.921	1.48	0.2062
Error	36	1321677697.407		
Corrected Total	43	1701508092.328		

R-Square	C.V.	Mg Mean
0.223232	22.95897	26391.205227

Source	DF	Type I SS	F Value	Pr > F
METH	7	379830394.921	1.48	0.2062
Source	DF	Type III SS	F Value	Pr > F
METH	7	379830394.921	1.48	0.2062

TABLE IV : Mg - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	23658.62	0.74	0.74	0.00
A1	2	8	30279.13	1200.49	0.87	0.17
A1	3	7	25550.29	1759.77	0.90	0.36
A1	4	5	26271.20	893.14	0.85	0.19
A1	7	3	23346.67	196.30	0.75	0.00
A1	8	3	25000.00	0.00		
A1	9	2	29900.00	282.84	1.00	1.00
A1	10	3	32123.33	119.84	0.88	0.33
A2	1	31	3037.35	1113.90	0.64	0.00
A2	2	9	2702.85	214.09	0.80	0.02
A2	3	7	2139.70	199.34	0.90	0.35
A2	4	5	2441.80	125.80	0.78	0.05
A2	5	3	3000.00	155.89	0.75	0.00
A2	6	1	2250.00	91.65	0.96	0.64
A2	7	3	2610.00	0.00		
A2	8	3	2240.00	0.00		
A2	9	2	2200.00	282.84	1.00	1.00
A2	10	8	2776.88	685.09	0.92	0.41
A3	4	5	2428.60	76.20	0.80	0.09
B1	1	13	3329.90	355.72	0.91	0.18
B1	2	8	3153.75	376.05	0.98	0.93
B1	3	7	2305.57	92.03	0.88	0.22
B1	4	5	2466.80	99.95	0.92	0.56
B1	7	3	2860.00	0.00		
B1	8	3	5268.67	155.39	0.97	0.66
B1	9	2	4300.00	0.00		
B1	10	3	4515.00	29.45	0.75	0.00
B2	1	13	244.31	31.86	0.74	0.00
B2	2	8	217.84	37.26	0.86	0.13
B2	3	7	196.25	10.65	0.92	0.50
B2	4	5	215.20	6.10	0.87	0.25
B2	7	3	330.00	0.00		
B2	8	3	381.42	13.72	0.88	0.33
B2	9	2	500.00	0.00		
B2	10	3	418.00	0.00		

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 5 and 3 replicates.

To calculate the total number of replicates for the method-grouped laboratories,

SAS counts the number of reported results.

So, the "repl" values in this table for methods 2 and 1 are different from the real number of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 36713269

Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

General Linear Models Procedure				
METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
8 - 10	-23031	-7123	8785	
8 - 2	-18469	-5279	7911	
8 - 9	-22686	-4900	12886	
8 - 4	-15500	-1271	12957	
8 - 3	-13995	-550	12894	
8 - 1	-11138	1341	13821	
8 - 7	-14255	1653	17561	
1 - 10	-20944	-8465	4014	
1 - 2	-15375	-6621	2134	
1 - 9	-21040	-6241	8557	
1 - 4	-12865	-2613	7640	
1 - 3	-11025	-1892	7242	
1 - 8	-13821	-1341	11138	
1 - 7	-12167	312	12791	
7 - 10	-24685	-8777	7131	
7 - 2	-20123	-6932	6258	
7 - 9	-24339	-6553	11232	
7 - 4	-17153	-2925	11304	
7 - 3	-15648	-2204	11241	
7 - 8	-17561	-1653	14255	
7 - 1	-12791	-312	12167	

General Linear Models Procedure				
METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
10 - 2	-11346	1844	15034	
10 - 9	-15562	2223	20009	
10 - 4	-8376	5852	20081	
10 - 3	-6872	6573	20018	
10 - 8	-8785	7123	23031	
10 - 1	-4014	8465	20944	
10 - 7	-7131	8777	24685	
2 - 10	-15034	-1844	11346	
2 - 9	-15024	379	15782	
2 - 4	-7099	4008	15115	
2 - 3	-5355	4729	14812	
2 - 8	-7911	5279	18469	
2 - 1	-2134	6621	15375	
2 - 7	-6258	6932	20123	
9 - 10	-20009	-2223	15562	
9 - 2	-15782	-379	15024	
9 - 4	-12672	3629	19930	
9 - 3	-11272	4350	19971	
9 - 8	-12886	4900	22686	
9 - 1	-8557	6241	21040	
9 - 7	-11232	6553	24339	
4 - 10	-20081	-5852	8376	
4 - 2	-15115	-4008	7099	
4 - 9	-19930	-3629	12672	
4 - 3	-10687	721	12129	
4 - 8	-12957	1271	15500	
4 - 1	-7640	2613	12865	
4 - 7	-11304	2925	17153	
3 - 10	-20018	-6573	6872	
3 - 2	-14812	-4729	5355	
3 - 9	-19971	-4350	11272	
3 - 4	-12129	-721	10687	
3 - 8	-12894	550	13995	
3 - 1	-7242	1892	11025	
3 - 7	-11241	2204	15648	

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 10 1 10 2 3 4 5 6 7 8 9

Number of observations in by group = 72

Dependent Variable: Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	9	7549183.62862	1.26	0.2775
Error	62	41322441.18468		
Corrected Total	71	48871624.81330		

R-Square C.V. Mg Mean

0.154470 29.67406 2751.1876389

Source	DF	Type I SS	F Value	Pr > F
METH	9	7549183.62862	1.26	0.2775
Source	DF	Type III SS	F Value	Pr > F
METH	9	7549183.62862	1.26	0.2775

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 62 MSE= 666491
Critical Value of Studentized Range= 4.641

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	Limit					Limit
1 - 5	-1582.4		37.4		1657.2	
1 - 10	-801.9		260.5		1322.8	
1 - 2	-679.9		334.5		1348.9	
1 - 7	-1192.4		427.4		2047.2	
1 - 4	-695.5		595.6		1886.6	
1 - 6	-1934.5		787.4		3509.2	
1 - 8	-822.4		797.4		2417.2	
1 - 9	-1117.1		837.4		2791.8	
1 - 3	-223.4		897.6		2018.7	
5 - 1	-1657.2		-37.4		1582.4	
5 - 10	-1590.5		223.1		2036.8	
5 - 2	-1488.8		297.2		2083.1	
5 - 7	-1797.3		390.0		2577.3	
5 - 4	-1398.2		558.2		2514.6	
5 - 6	-2343.4		750.0		3843.4	
5 - 8	-1427.3		760.0		2947.3	
5 - 9	-1645.5		800.0		3245.5	
5 - 3	-988.4		860.3		2708.9	
10 - 1	-1322.8		-260.5		801.9	
10 - 5	-2036.8		-223.1		1590.5	
10 - 2	-1227.7		74.0		1375.8	
10 - 7	-1646.8		166.9		1980.5	
10 - 4	-1192.2		335.1		1862.3	
10 - 6	-2314.6		526.9		3368.3	
10 - 8	-1276.8		536.9		2350.5	
10 - 9	-1541.0		576.9		2694.8	
10 - 3	-749.3		637.2		2023.7	
2 - 1	-1348.9		-334.5		679.9	
2 - 5	-2083.1		-297.2		1488.8	
2 - 10	-1375.8		-74.0		1227.7	
2 - 7	-1693.1		92.8		1878.8	
2 - 4	-1233.2		261.0		1755.3	
2 - 6	-2371.0		452.8		3276.7	
2 - 8	-1323.1		462.8		2248.8	
2 - 9	-1591.4		502.8		2597.1	
2 - 3	-786.9		563.1		1913.2	

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	9	3	9	3	9	3
9 - 3	-2087.6	60.3	-2087.6	60.3	2208.2	2208.2
3 - 1	-2018.7	-897.6	-2018.7	-897.6	223.4	223.4
3 - 5	-2708.9	-860.3	-2708.9	-860.3	988.4	988.4
3 - 10	-2023.7	-637.2	-2023.7	-637.2	749.3	749.3
3 - 2	-1913.2	-563.1	-1913.2	-563.1	786.9	786.9
3 - 7	-2318.9	-470.3	-2318.9	-470.3	1378.4	1378.4
3 - 4	-1870.7	-302.1	-1870.7	-302.1	1266.5	1266.5
3 - 6	-2974.2	-110.3	-2974.2	-110.3	2753.6	2753.6
3 - 8	-1948.9	-100.3	-1948.9	-100.3	1748.4	1748.4
3 - 9	-2208.2	-60.3	-2208.2	-60.3	2087.6	2087.6

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	7	3	7	3	7	3
7 - 1	-2047.2	-427.4	-2047.2	-427.4	1192.4	1192.4
7 - 5	-2577.3	-390.0	-2577.3	-390.0	1797.3	1797.3
7 - 10	-1980.5	-166.9	-1980.5	-166.9	1646.8	1646.8
7 - 2	-1878.8	-92.8	-1878.8	-92.8	1693.1	1693.1
7 - 4	-1788.2	168.2	-1788.2	168.2	2124.6	2124.6
7 - 6	-2733.4	360.0	-2733.4	360.0	3453.4	3453.4
7 - 8	-1817.3	370.0	-1817.3	370.0	2557.3	2557.3
7 - 9	-2035.5	410.0	-2035.5	410.0	2855.5	2855.5
7 - 3	-1378.4	470.3	-1378.4	470.3	2318.9	2318.9
4 - 1	-1886.6	-595.6	-1886.6	-595.6	695.5	695.5
4 - 5	-2514.6	-558.2	-2514.6	-558.2	1398.2	1398.2
4 - 10	-1862.3	-335.1	-1862.3	-335.1	1192.2	1192.2
4 - 2	-1755.3	-261.0	-1755.3	-261.0	1233.2	1233.2
4 - 7	-2124.6	-168.2	-2124.6	-168.2	1788.2	1788.2
4 - 6	-2742.8	191.8	-2742.8	191.8	3126.4	3126.4
4 - 8	-1754.6	201.8	-1754.6	201.8	2158.2	2158.2
4 - 9	-1999.6	241.8	-1999.6	241.8	2483.2	2483.2
4 - 3	-1266.5	302.1	-1266.5	302.1	1870.7	1870.7
6 - 1	-3509.2	-787.4	-3509.2	-787.4	1934.5	1934.5
6 - 5	-3843.4	-750.0	-3843.4	-750.0	2343.4	2343.4
6 - 10	-3368.3	-526.9	-3368.3	-526.9	2314.6	2314.6
6 - 2	-3276.7	-452.8	-3276.7	-452.8	2371.0	2371.0
6 - 7	-3453.4	-360.0	-3453.4	-360.0	2733.4	2733.4
6 - 4	-3126.4	-191.8	-3126.4	-191.8	2742.8	2742.8
6 - 8	-3083.4	10.0	-3083.4	10.0	3103.4	3103.4
6 - 9	-3231.0	50.0	-3231.0	50.0	3331.0	3331.0
6 - 3	-2753.6	110.3	-2753.6	110.3	2974.2	2974.2
8 - 1	-2417.2	-797.4	-2417.2	-797.4	822.4	822.4
8 - 5	-2947.3	-760.0	-2947.3	-760.0	1427.3	1427.3
8 - 10	-2350.5	-536.9	-2350.5	-536.9	1276.8	1276.8
8 - 2	-2248.8	-462.8	-2248.8	-462.8	1323.1	1323.1
8 - 7	-2557.3	-370.0	-2557.3	-370.0	1817.3	1817.3
8 - 4	-2158.2	-201.8	-2158.2	-201.8	1754.6	1754.6
8 - 6	-3103.4	-10.0	-3103.4	-10.0	3083.4	3083.4
8 - 9	-2405.5	40.0	-2405.5	40.0	2485.5	2485.5
8 - 3	-1748.4	100.3	-1748.4	100.3	1948.9	1948.9
9 - 1	-2791.8	-837.4	-2791.8	-837.4	1117.1	1117.1
9 - 5	-3245.5	-800.0	-3245.5	-800.0	1645.5	1645.5
9 - 10	-2694.8	-576.9	-2694.8	-576.9	1541.0	1541.0
9 - 2	-2597.1	-502.8	-2597.1	-502.8	1591.4	1591.4
9 - 7	-2855.5	-410.0	-2855.5	-410.0	2035.5	2035.5
9 - 4	-2483.2	-241.8	-2483.2	-241.8	1999.6	1999.6
9 - 6	-3331.0	-50.0	-3331.0	-50.0	3231.0	3231.0
9 - 8	-2485.5	-40.0	-2485.5	-40.0	2405.5	2405.5

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 73587.39
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '****'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
8	- 10	41.5	1465.9	753.7	753.7	1465.9
8	- 9	172.4	1764.9	968.7	968.7	1764.9
8	- 1	1380.1	2497.5	1938.8	1938.8	2497.5
8	- 2	1524.4	2705.4	2114.9	2114.9	2705.4
8	- 7	1696.5	3120.9	2408.7	2408.7	3120.9
8	- 4	2164.9	3438.9	2801.9	2801.9	3438.9
8	- 3	2361.2	3565.0	2963.1	2963.1	3565.0
10	- 8	-1465.9	-41.5	-753.7	-753.7	-41.5
10	- 9	-581.3	1011.3	215.0	215.0	1011.3
10	- 1	626.4	1743.8	1185.1	1185.1	1743.8
10	- 2	770.7	1951.8	1361.2	1361.2	1951.8
10	- 7	942.8	2367.2	1655.0	1655.0	2367.2
10	- 4	1411.2	2685.2	2048.2	2048.2	2685.2
10	- 3	1607.5	2811.4	2209.4	2209.4	2811.4
9	- 8	-1764.9	-172.4	-968.7	-968.7	-172.4
9	- 10	-1011.3	581.3	-215.0	-215.0	581.3
9	- 1	307.6	1632.6	970.1	970.1	1632.6
9	- 2	456.7	1835.8	1146.2	1146.2	1835.8
9	- 7	643.7	2236.3	1440.0	1440.0	2236.3
9	- 4	1103.4	2563.0	1833.2	1833.2	2563.0
9	- 3	1295.1	2693.8	1994.4	1994.4	2693.8
1	- 8	-2497.5	-1380.1	-1938.8	-1938.8	-1380.1
1	- 10	-1743.8	-626.4	-1185.1	-1185.1	-626.4
1	- 9	-1632.6	-307.6	-970.1	-970.1	-307.6
1	- 2	-215.8	568.1	176.2	176.2	568.1
1	- 7	-88.8	1028.6	469.9	469.9	1028.6
1	- 4	404.1	1322.1	863.1	863.1	1322.1
1	- 3	615.4	1433.3	1024.3	1024.3	1433.3
2	- 8	-2705.4	-1524.4	-2114.9	-2114.9	-1524.4
2	- 10	-1951.8	-770.7	-1361.2	-1361.2	-770.7
2	- 9	-1835.8	-456.7	-1146.2	-1146.2	-456.7
2	- 1	-568.1	215.8	-176.2	-176.2	215.8
2	- 7	-296.8	884.3	293.8	293.8	884.3
2	- 4	189.7	1184.2	686.9	686.9	1184.2
2	- 3	396.7	1299.6	848.2	848.2	1299.6

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 10 2 3 4 7 8 9

Number of observations in by group = 44

Dependent Variable: Mg				Mg Mean	
Source	DF	Sum of Squares	F Value	Pr > F	
Model	7	29148582.2630	56.59	0.0001	
Error	36	2649146.2084			
Corrected Total	43	31797728.4714			
R-Square				C.V.	
	0.916688	8.316371	3261.8790909		
				Pr > F	
Source	DF	Type I SS	F Value	Pr > F	
METH	7	29148582.2630	56.59	0.0001	
				Pr > F	
Source	DF	Type III SS	F Value	Pr > F	
METH	7	29148582.2630	56.59	0.0001	

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 10 2 3 4 7 8 9

Number of observations in by group = 44

Dependent Variable: Mg

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	302655.127169	67.36	0.0001
Error	36	23106.061895		
Corrected Total	43	325761.189064		
R-Square C.V. Mg Mean				
		9.481606	267.19590909	
Source	DF	Type I SS	F Value	Pr > F
METH	7	302655.127169	67.36	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	7	302655.127169	67.36	0.0001

SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
7 - 8	-3120.9	-2408.7	-1696.5	***
7 - 10	-2367.2	-1655.0	-942.8	***
7 - 9	-2236.3	-1440.0	-643.7	***
7 - 1	-1028.6	-469.9	88.8	
7 - 2	-884.3	-293.8	296.8	
7 - 4	-243.8	393.2	1030.2	
7 - 3	-47.5	554.4	1156.4	
4 - 8	-3438.9	-2801.9	-2164.9	***
4 - 10	-2685.2	-2048.2	-1411.2	***
4 - 9	-2563.0	-1833.2	-1103.4	***
4 - 1	-1322.1	-863.1	-404.1	***
4 - 2	-1184.2	-686.9	-189.7	***
4 - 7	-1030.2	-393.2	243.8	
4 - 3	-349.5	161.2	672.0	
3 - 8	-3565.0	-2963.1	-2361.2	***
3 - 10	-2811.4	-2209.4	-1607.5	***
3 - 9	-2693.8	-1994.4	-1295.1	***
3 - 1	-1433.3	-1024.3	-615.4	***
3 - 2	-1299.6	-848.2	-396.7	***
3 - 7	-1156.4	-554.4	47.5	
3 - 4	-672.0	-161.2	349.5	

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mg

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 36 MSE= 641.8351
Critical Value of Studentized Range= 4.547

Comparisons significant at the 0.05 level are indicated by '***'.

			Simultaneous			Simultaneous		
			Lower	Difference	Upper	Lower	Difference	Upper
			Limit	Means	Limit	Limit	Means	Limit
METH	Comparison	Confidence	Limit	Means	Limit	Limit	Means	Limit
9	- 10	7.635	82.000	156.365	***	346.567	-282.165	-217.763
9	- 8	44.215	118.580	192.945	***	-255.315	-200.165	-145.015
9	- 7	95.635	170.000	244.365	***	-218.735	-163.585	-108.435
9	- 1	193.820	255.695	317.571	***	-167.315	-112.165	-57.015
9	- 2	217.763	282.165	346.567	***	-63.075	-26.470	10.136
9	- 4	216.644	284.800	352.956	***	-43.806	2.635	49.076
9	- 3	238.439	303.754	369.070	***	-20.572	21.589	63.750
10	- 9	-156.365	-82.000	-7.635	***	-352.956	-284.800	-216.644
10	- 8	-29.934	36.580	103.094	***	-262.292	-202.800	-143.308
10	- 7	21.486	88.000	154.514	***	-225.712	-166.220	-106.728
10	- 1	121.518	173.695	225.873	***	-174.292	-114.800	-55.308
10	- 2	145.015	200.165	255.315	***	-71.973	-29.105	13.764
10	- 4	143.308	202.800	262.292	***	-49.076	-2.635	43.806
10	- 3	165.540	221.754	277.969	***	-28.745	18.954	66.654
8	- 9	-192.945	-118.580	-44.215	***	-369.070	-303.754	-238.439
8	- 10	-103.094	-36.580	29.934	***	-277.969	-221.754	-165.540
8	- 7	-15.094	51.420	117.934	***	-241.389	-185.174	-128.960
8	- 1	84.938	137.115	189.293	***	-189.969	-133.754	-77.540
8	- 2	108.435	163.585	218.735	***	-86.249	-48.059	-9.869
8	- 4	106.728	166.220	225.712	***	-63.750	-21.589	20.572
8	- 3	128.960	185.174	241.389	***	-66.654	-18.954	28.745
7	- 9	-244.365	-170.000	-95.635	***	-369.070	-303.754	-238.439
7	- 10	-154.514	-88.000	-21.486	***	-277.969	-221.754	-165.540
7	- 8	-117.934	-51.420	15.094	***	-241.389	-185.174	-128.960
7	- 1	33.518	85.695	137.873	***	-189.969	-133.754	-77.540
7	- 2	57.015	112.165	167.315	***	-86.249	-48.059	-9.869
7	- 4	55.308	114.800	174.292	***	-63.750	-21.589	20.572
7	- 3	77.540	133.754	189.969	***	-66.654	-18.954	28.745
1	- 9	-317.571	-255.695	-193.820	***	-369.070	-303.754	-238.439
1	- 10	-225.873	-173.695	-121.518	***	-277.969	-221.754	-165.540
1	- 8	-189.293	-137.115	-84.938	***	-241.389	-185.174	-128.960
1	- 7	-137.873	-85.695	-33.518	***	-189.969	-133.754	-77.540
1	- 2	-10.136	26.470	63.075	***	-86.249	-48.059	-9.869
1	- 4	-13.764	29.105	71.973	***	-63.750	-21.589	20.572
1	- 3	9.869	48.059	86.249	***	-66.654	-18.954	28.745

General Linear Models Procedure

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

Confidence

Limit

Simultaneous

Lower

Confidence

Limit

Simultaneous

Upper

25. Na

units: mg/kg
optional for all samples
reported by 12 laboratories

TABLE I : Na - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1			
2	A1 A2 B1 B2	5	N
3	A1 A2 B1 B2	4	N
4			
5			
6	A1 A2 B1 B2	3	N
7	A1 A2 B1 B2	3	N
8			
9	A1 A2 B1 B2	3	R
10		1 not reported	N
11	B2		
12			
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	A2:3 B2:2 A1 B1: 4	N
16	A1 A2 B1 B2	3	N/R
17			
18	A1 A2 B1 B2	3	N/R
19			
20	A2	1 not reported	N
21			
22	A2	3	R

(*) R = aqua regia

TABLE II : Na - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	dry ashing
5	digestion, HNO3 under pressure
6	extractant HF
7	combustion, HClO4 + H2SO4
*	minéralisation
*	wet ashing
*	extractant 0.2N BaCl2 at pH 8.1
10	ignition, HF + HClO4
11	unspecified
12	

* not applicable

TABLE IIIa : Na - Descriptive statistics

sample A1 - national methods

labcode	method	repl	min	mean	max	std	cv
1							
2							
3	2	5	136.23	168.74	175.64	0.46	0.27
4	3	4	93.40	98.73	108.00	6.99	7.01
5							
6							
7	11	3	8017.00	8051.00	8088.00	35.60	0.44
8	12	3	831.00	897.67	962.00	77.04	8.58
9							
10							
11							
12							
13							
14	4	5	157.00	207.00	231.00	29.76	14.38
15	2	4	102.40	109.55	118.40	6.94	6.33
16	5	3	712.00	725.00	740.00	14.11	1.95
17							
18	3	3	215.39	217.42	220.47	2.69	1.24
19							
20							
21							
22							

sample B1 - national methods

labcode	method	repl	min	mean	max	std	cv
1							
2							
3	2	5	77.02	84.83	93.34	5.29	6.23
4	3	4	50.80	52.10	53.80	1.37	2.63
5							
6							
7	11	3	7120.00	7293.00	7466.00	173.00	2.37
8	12	3	491.00	2049.00	5127.00	2665.69	130.10
9							
10							
11							
12							
13							
14	4	5	162.00	171.20	177.00	5.63	3.29
15	2	4	53.10	69.90	91.80	18.84	24.09
16	5	3	281.00	282.67	285.00	2.08	0.74
17							
18	3	3	85.16	90.47	93.89	4.66	5.16
19							
20							
21							
22							

sample A2 - national method

labcode	method	repl	min	mean	max	std	cv
1							
2							
3	2	5	71.96	80.46	91.73	8.05	10.00
4	3	4	52.34	53.14	53.90	0.70	1.32
5							
6							
7	11	3	681.00	807.00	959.00	140.81	17.45
8	12	3	301.00	314.79	332.00	15.78	5.01
9							
10							
11							
12							
13							
14	4	5	109.00	118.80	128.00	8.87	7.47
15	A3	5	110.00	117.80	123.00	6.26	5.32
16	2	3	47.90	52.03	55.80	3.96	7.62
17	5	3	167.00	173.33	177.00	5.51	3.18
18							
19	3	3	78.37	80.15	82.29	1.99	2.48
20							
21	6	1		66.00			
22							

sample B2 - national method

labcode	method	repl	min	mean	max	std	cv
1							
2							
3	2	5	8.05	17.24	20.92	2.53	14.67
4	3	4	35.10	35.73	36.90	0.85	2.38
5							
6							
7	11	3	1071.00	1145.67	1193.00	65.43	5.71
8	12	3	108.90	108.97	109.00	0.06	0.05
9							
10							
11							
12							
13							
14	4	5	31.60	32.66	33.60	0.87	2.68
15	2	2	19.40	19.90	20.40	0.71	3.55
16	5	3	49.00	59.00	64.00	8.66	14.68
17							
18	3	3	49.39	52.32	55.66	3.15	6.03
19							
20							
21							
22							

TABLE IIIb : Na - Descriptive statistics

sample A1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	157.00	164.33	174.00	8.74	5.32
11						
12						
13						
14						
15						
16	3	129.00	132.33	136.00	3.51	2.65
17						
18	3	353.57	368.47	392.18	20.76	5.63
19						
20						
21						
22						

sample A2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	92.00	100.17	110.00	9.12	9.10
11						
12						
13						
14						
15						
16	3	15.00	25.33	36.00	10.50	41.46
17						
18	3	206.53	208.34	211.97	3.14	1.51
19						
20						
21						
22	3	60.00	62.33	65.00	2.52	4.04

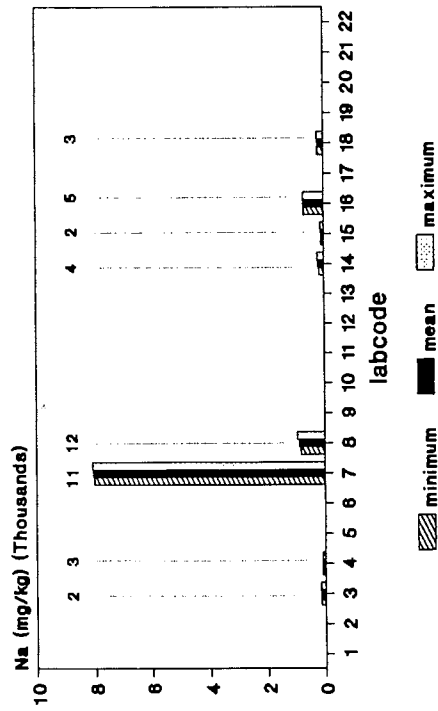
sample B1 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	71.00	74.67	77.00	3.22	4.31
11						
12						
13						
14						
15						
16	3	64.00	66.67	69.00	2.52	3.78
17						
18	3	198.94	234.47	291.31	49.74	21.21
19						
20						
21						
22						

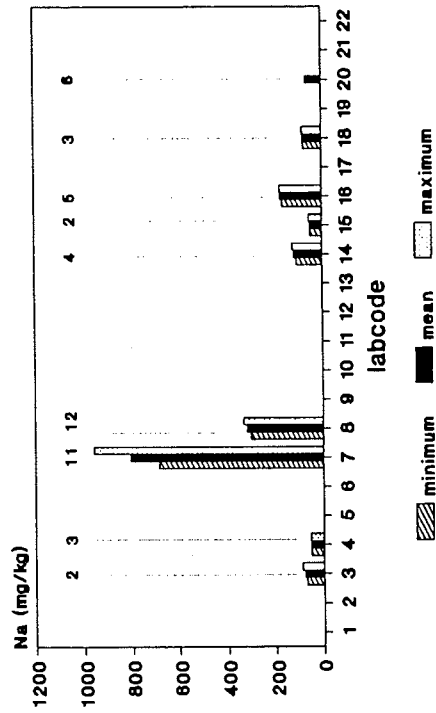
sample B2 - reference method

labcode	repl	minimum	mean	maximum	std	cv
1						
2						
3						
4						
5						
6						
7						
8						
9						
10	3	14.50	15.50	17.50	1.73	11.18
11	1		200.00			
12						
13						
14						
15						
16	3	8.00	9.67	13.00	2.89	29.86
17						
18	3	122.45	138.31	146.74	13.74	9.94
19						
20						
21						
22						

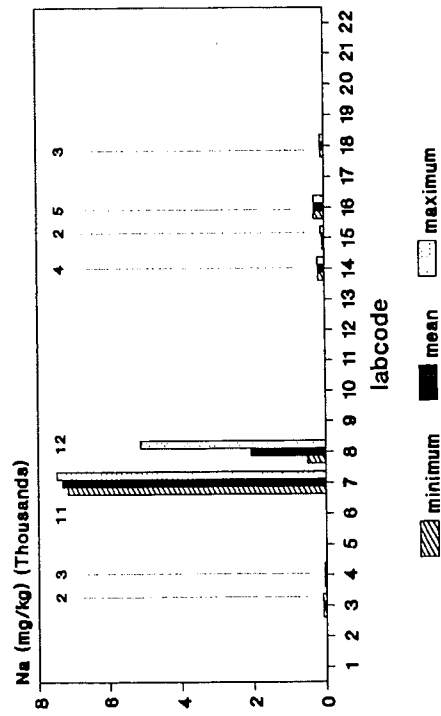
Na sample A1
national methods



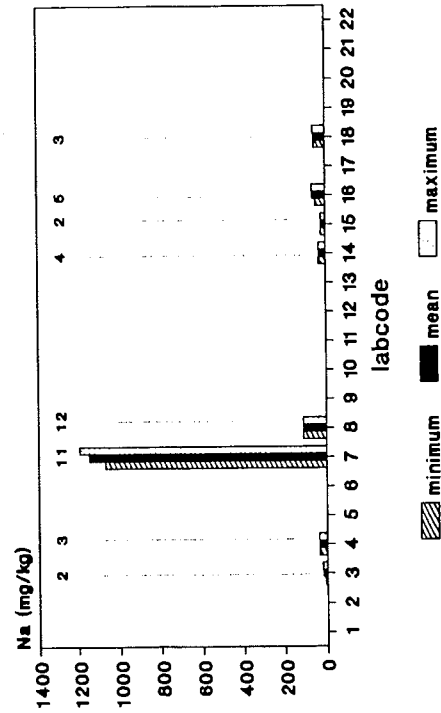
Na sample A2
national methods



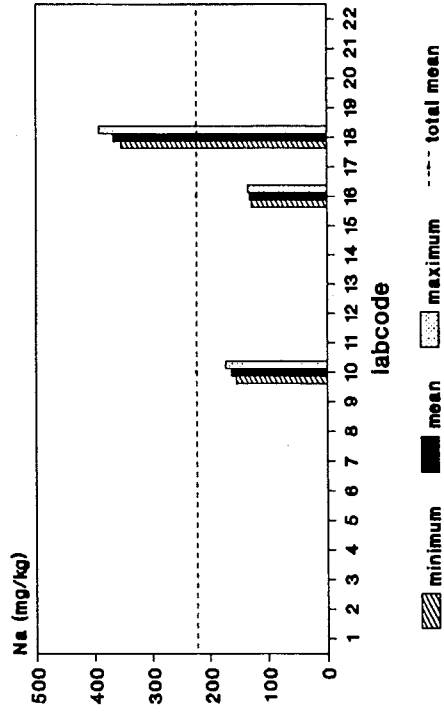
Na sample B1
national methods



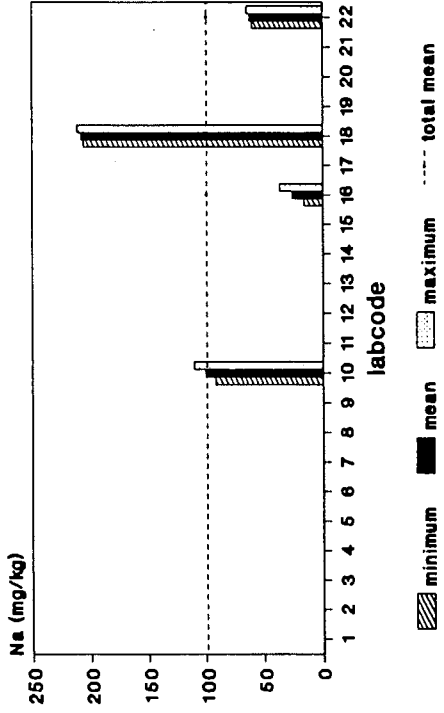
Na sample B2
national methods



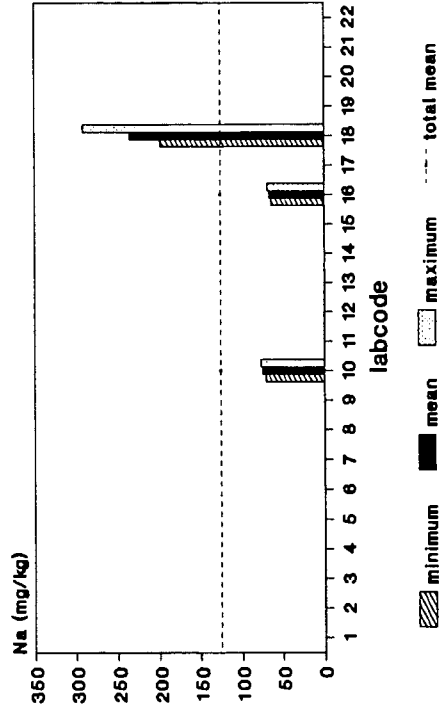
Na sample A1
reference method



Na sample A2
reference method



Na sample B1
reference method



Na sample B2
reference method

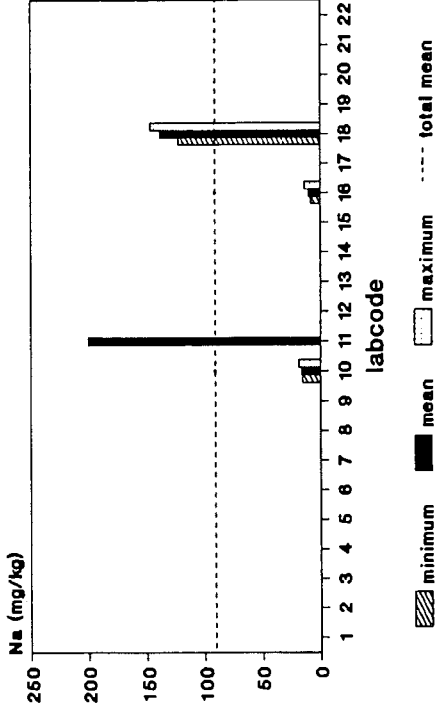


TABLE V :

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 7 1 11 12 2 3 4 5

Number of observations in by group = 35

Dependent Variable: Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	166871651.443	5381.94	0.0001
Error	28	144694.045		
Corrected Total	34	167016345.488		

R-Square C.V. Na Mean

0.999134 7.463831 963.12914286

Source	DF	Type I SS	F Value	Pr > F
METH	6	166871651.443	5381.94	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	166871651.443	5381.94	0.0001

TABLE IV : Na - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	221.71	111.52	0.76	0.01
A1	2	5	121.39	27.14	0.75	0.03
A1	3	7	150.17	63.13	0.72	0.01
A1	4	5	207.00	29.76	0.83	0.14
A1	5	3	725.00	14.11	0.99	0.76
A1	11	3	8051.00	35.60	1.00	0.86
A1	12	3	897.67	77.04	0.96	0.62
A2	1	12	99.04	71.74	0.86	0.05
A2	2	4	59.14	14.58	0.83	0.15
A2	3	7	64.72	14.49	0.73	0.01
A2	4	5	118.80	8.87	0.86	0.24
A2	5	3	173.33	5.51	0.82	0.17
A2	6	1	66.00			
A2	11	3	807.00	140.81	0.97	0.69
A2	12	3	314.79	15.78	0.97	0.64
A3	4	5	117.80	6.26	0.78	0.06
B1	1	9	125.27	85.69	0.73	0.00
B1	2	5	72.89	16.04	0.96	0.81
B1	3	7	68.55	20.71	0.75	0.01
B1	4	5	171.20	5.63	0.90	0.42
B1	5	3	282.67	2.08	0.92	0.46
B1	11	3	7293.00	173.00	1.00	0.99
B1	12	3	2049.00	2665.69	0.76	0.01
B2	1	10	69.04	75.37	0.77	0.01
B2	2	3	19.01	1.62	0.96	0.60
B2	3	7	42.84	9.08	0.79	0.03
B2	4	5	32.66	0.87	0.91	0.47
B2	5	3	59.00	8.66	0.75	0.00
B2	11	3	1145.67	65.43	0.87	0.29
B2	12	3	108.97	0.06	0.75	0.00

Remark:

Labcode 3 reported mean values for 5 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for method 2 are different from the real number
of replicates.

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
3 - 4	-190.36	-56.83	76.69	-190.36	-56.83	76.69
3 - 2	-104.74	28.78	162.30	-104.74	28.78	162.30
2 - 11	-8096.14	-7929.61	-7763.08	-8096.14	-7929.61	-7763.08
2 - 12	-942.81	-776.28	-609.75	-942.81	-776.28	-609.75
2 - 5	-770.14	-603.61	-437.08	-770.14	-603.61	-437.08
2 - 1	-227.52	-100.32	26.87	-227.52	-100.32	26.87
2 - 4	-229.83	-85.61	58.61	-229.83	-85.61	58.61
2 - 3	-162.30	-28.78	104.74	-162.30	-28.78	104.74

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSE= 5167.644
Critical Value of Studentized Range= 4.486

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 12	6967.14	7153.33	7339.52	7339.52	***	***
11 - 5	7139.81	7326.00	7512.19	7512.19	***	***
11 - 1	7677.27	7829.29	7981.31	7981.31	***	***
11 - 4	7677.47	7844.00	8010.53	8010.53	***	***
11 - 3	7743.47	7900.83	8058.19	8058.19	***	***
11 - 2	7763.08	7929.61	8096.14	8096.14	***	***
12 - 11	-7339.52	-7153.33	-6967.14	-6967.14	***	***
12 - 5	-13.52	172.67	358.86	358.86	***	***
12 - 1	523.93	675.95	827.98	827.98	***	***
12 - 4	524.13	690.67	857.20	857.20	***	***
12 - 3	590.14	747.50	904.86	904.86	***	***
12 - 2	609.75	776.28	942.81	942.81	***	***
5 - 11	-7512.19	-7326.00	-7139.81	-7139.81	***	***
5 - 12	-358.86	-172.67	13.52	13.52	***	***
5 - 1	351.27	503.29	655.31	655.31	***	***
5 - 4	351.47	518.00	684.53	684.53	***	***
5 - 3	417.47	574.83	732.19	732.19	***	***
5 - 2	437.08	603.61	770.14	770.14	***	***
1 - 11	-7981.31	-7829.29	-7677.27	-7677.27	***	***
1 - 12	-827.98	-675.95	-523.93	-523.93	***	***
1 - 5	-655.31	-503.29	-351.27	-351.27	***	***
1 - 4	-112.48	14.71	141.90	141.90	***	***
1 - 3	-43.37	71.55	186.46	186.46	***	***
1 - 2	-26.87	100.32	227.52	227.52	***	***
4 - 11	-8010.53	-7844.00	-7677.47	-7677.47	***	***
4 - 12	-857.20	-690.67	-524.13	-524.13	***	***
4 - 5	-684.53	-518.00	-351.47	-351.47	***	***
4 - 1	-141.90	-14.71	112.48	112.48	***	***
4 - 3	-76.69	56.83	190.36	190.36	***	***
4 - 2	-58.61	85.61	229.83	229.83	***	***
3 - 11	-8058.19	-7900.83	-7743.47	-7743.47	***	***
3 - 12	-904.86	-747.50	-590.14	-590.14	***	***
3 - 5	-732.19	-574.83	-417.47	-417.47	***	***
3 - 1	-186.46	-71.55	43.37	43.37	***	***

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 30 MSE= 3301.053
Critical Value of Studentized Range= 4.601

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit			
11	- 12	339.57	492.21	644.85	***	
11	- 5	481.03	633.67	786.30	***	
11	- 4	551.68	688.20	824.72	***	
11	- 1	587.29	707.96	828.63	***	
11	- 6	525.14	741.00	956.86	***	
11	- 3	613.28	742.28	871.28	***	
11	- 2	605.08	747.86	890.64	***	
12	- 11	-644.85	-492.21	-339.57	***	
12	- 5	-11.18	141.46	294.09	***	
12	- 4	59.47	195.99	332.51	***	
12	- 1	95.08	215.75	336.42	***	
12	- 6	32.93	248.79	464.65	***	
12	- 3	121.07	250.07	379.07	***	
12	- 2	112.87	255.65	398.43	***	
5	- 11	-786.30	-633.67	-481.03	***	
5	- 12	-294.09	-141.46	11.18		
5	- 4	-81.99	54.53	191.06		
5	- 1	-46.38	74.29	194.96		
5	- 6	-108.53	107.33	323.19		
5	- 3	-20.38	108.62	237.62		
5	- 2	-28.58	114.19	256.97		
4	- 11	-824.72	-688.20	-551.68	***	
4	- 12	-332.51	-195.99	-59.47	***	
4	- 5	-191.06	-54.53	81.99		
4	- 1	-79.75	19.76	119.26		
4	- 6	-151.98	52.80	257.58		
4	- 3	-55.38	54.08	163.54		
4	- 2	-65.74	59.66	185.06		
1	- 11	-828.63	-707.96	-587.29	***	
1	- 12	-336.42	-215.75	-95.08	***	
1	- 5	-194.96	-74.29	46.38		
1	- 4	-119.26	-19.76	79.75		
1	- 6	-161.53	33.04	227.62		
1	- 3	-54.58	34.33	123.23		
1	- 2	-68.03	39.90	147.83		

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 11 12 2 3 4 5

Number of observations in by group = 35

Dependent Variable: Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	144031543.831	46.89	0.0001
Error	28	14334181.663		
Corrected Total	34	158365725.494		

R-Square C.V. Na Mean

0.909487 78.99391 905.76142857

Source	DF	Type I SS	F Value	Pr > F
METH	6	144031543.831	46.89	0.0001
Source	DF	Type III SS <td>F Value <td>Pr > F</td> </td>	F Value <td>Pr > F</td>	Pr > F
METH	6	144031543.831	46.89	0.0001

SAMPLE=A2

General Linear Models Procedure

Simultaneous Lower Confidence Limit

Simultaneous Upper Confidence Limit

Difference Between Means

Confidence Limit

Confidence Limit

Confidence Limit

Confidence Limit

Confidence Limit

Confidence Limit

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SAMPLE=B1

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
2 - 1	-1318.3	-52.4	1213.6	-1318.3	-52.4	1213.6
2 - 3	-1324.6	4.3	1333.3	-1324.6	4.3	1333.3
3 - 11	-8790.7	-7224.5	-5658.2	-8790.7	-7224.5	-5658.2
3 - 12	-3546.7	-1980.5	-414.2	-3546.7	-1980.5	-414.2
3 - 5	-1780.3	-214.1	1352.1	-1780.3	-214.1	1352.1
3 - 4	-1431.6	-102.7	1226.3	-1431.6	-102.7	1226.3
3 - 1	-1200.5	-56.7	1087.1	-1200.5	-56.7	1087.1
3 - 2	-1333.3	-4.3	1324.6	-1333.3	-4.3	1324.6

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSE= 511935.1
Critical value of Studentized Range= 4.486

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
11 - 12	3390.8	5244.0	7097.2	3390.8	5244.0	7097.2
11 - 5	5157.2	7010.3	8863.5	5157.2	7010.3	8863.5
11 - 4	5464.3	7121.8	8779.3	5464.3	7121.8	8779.3
11 - 1	5654.6	7167.7	8680.8	5654.6	7167.7	8680.8
11 - 2	5562.6	7220.1	8877.6	5562.6	7220.1	8877.6
11 - 3	5658.2	7224.5	8790.7	5658.2	7224.5	8790.7
12 - 11	-7097.2	-5244.0	-3390.8	-7097.2	-5244.0	-3390.8
12 - 5	-86.8	1766.3	3619.5	-86.8	1766.3	3619.5
12 - 4	220.3	1877.8	3535.3	220.3	1877.8	3535.3
12 - 1	410.6	1923.7	3436.8	410.6	1923.7	3436.8
12 - 2	318.6	1976.1	3633.6	318.6	1976.1	3633.6
12 - 3	414.2	1980.5	3546.7	414.2	1980.5	3546.7
5 - 11	-8863.5	-7010.3	-5157.2	-8863.5	-7010.3	-5157.2
5 - 12	-3619.5	-1766.3	86.8	-3619.5	-1766.3	86.8
5 - 4	-1546.1	111.5	1769.0	-1546.1	111.5	1769.0
5 - 1	-1355.7	157.4	1670.5	-1355.7	157.4	1670.5
5 - 2	-1447.7	209.8	1867.3	-1447.7	209.8	1867.3
5 - 3	-1352.1	214.1	1780.3	-1352.1	214.1	1780.3
4 - 11	-8779.3	-7121.8	-5464.3	-8779.3	-7121.8	-5464.3
4 - 12	-3535.3	-1877.8	-220.3	-3535.3	-1877.8	-220.3
4 - 5	-1769.0	-111.5	1546.1	-1769.0	-111.5	1546.1
4 - 1	-1220.0	45.9	1311.9	-1220.0	45.9	1311.9
4 - 2	-1337.1	98.3	1533.8	-1337.1	98.3	1533.8
4 - 3	-1226.3	102.7	1431.6	-1226.3	102.7	1431.6
1 - 11	-8680.8	-7167.7	-5654.6	-8680.8	-7167.7	-5654.6
1 - 12	-3436.8	-1923.7	-410.6	-3436.8	-1923.7	-410.6
1 - 5	-1670.5	-157.4	1355.7	-1670.5	-157.4	1355.7
1 - 4	-1311.9	-45.9	1220.0	-1311.9	-45.9	1220.0
1 - 2	-1213.6	52.4	1318.3	-1213.6	52.4	1318.3
1 - 3	-1087.1	56.7	1200.5	-1087.1	56.7	1200.5
2 - 11	-8877.6	-7220.1	-5562.6	-8877.6	-7220.1	-5562.6
2 - 12	-3633.6	-1976.1	-318.6	-3633.6	-1976.1	-318.6
2 - 5	-1867.3	-209.8	1447.7	-1867.3	-209.8	1447.7
2 - 4	-1533.8	-98.3	1337.1	-1533.8	-98.3	1337.1

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Na

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 27 MSE= 2234.627
Critical Value of Studentized Range= 4.498

Comparisons significant at the 0.05 level are indicated by '***'.

			Simultaneous			Simultaneous		
			Lower		Difference Between Means	Upper		
			Confidence			Confidence		
			Limit			Limit		
			Comparison			Comparison		
			METH			METH		
			Comparison			Comparison		
11	-	12	11	-	12	1036.70	1159.47	***
11	-	1	11	-	1	1076.62	1175.60	***
11	-	5	11	-	5	1086.67	1209.44	***
11	-	3	11	-	3	1102.83	1206.59	***
11	-	4	11	-	4	1113.01	1222.81	***
11	-	2	11	-	2	1126.65	1249.42	***
12	-	11	12	-	11	-1036.70	-913.93	***
12	-	1	12	-	1	39.92	138.90	
12	-	5	12	-	5	49.97	172.74	
12	-	3	12	-	3	66.13	169.89	
12	-	4	12	-	4	76.31	186.11	
12	-	2	12	-	2	89.95	212.72	
1	-	11	1	-	11	-1076.62	-977.65	***
1	-	12	1	-	12	-39.92	59.05	
1	-	5	1	-	5	10.04	109.02	
1	-	3	1	-	3	26.20	100.30	
1	-	4	1	-	4	36.38	118.74	
1	-	2	1	-	2	50.03	149.01	
5	-	11	5	-	11	-1086.67	-963.90	***
5	-	12	5	-	12	-49.97	72.80	
5	-	1	5	-	1	-10.04	88.94	
5	-	3	5	-	3	16.16	119.92	
5	-	4	5	-	4	26.34	136.15	
5	-	2	5	-	2	39.99	162.76	
3	-	11	3	-	11	-1102.83	-999.07	***
3	-	12	3	-	12	-66.13	37.63	
3	-	1	3	-	1	-26.20	47.90	
3	-	5	3	-	5	-16.16	87.60	
3	-	4	3	-	4	10.18	98.22	
3	-	2	3	-	2	23.83	127.58	
4	-	11	4	-	11	-1113.01	-1003.20	***
4	-	12	4	-	12	-76.31	33.50	
4	-	1	4	-	1	-26.38	45.97	
4	-	5	4	-	5	-36.34	83.47	

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 7 1 11 12 2 3 4 5

Number of observations in by group. = 34

Dependent Variable: Na

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	3270115.31699	243.90	0.0001
Error	27	60334.93945		
Corrected Total	33	3330450.25644		

R-Square C.V. Na Mean

0.981884 31.19933 151.51558824

Source	DF	Type I SS	F Value	Pr > F
METH	6	3270115.31699	243.90	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	3270115.31699	243.90	0.0001

TABLE III : S - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	20.00	20.00	20.00	0.00	0.00
2							
3	2	5	371.25	376.71	380.23	4.17	1.11
4	3	4	307.00	338.25	370.00	28.49	8.42
5							
6							
7	4	3	406.00	411.00	416.00	5.00	1.22
8							
9							
10	1	3	6.00	7.67	10.00	2.08	27.15
11	8	3	240.00	263.33	280.00	20.82	7.91
12							
13							
14							
15	6	2	340.50	340.50	340.50	0.00	0.00
16	7	3	489.00	501.00	509.00	10.58	2.11
17							
18							
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	1790.00	1868.67	1910.00	66.58	3.57
2							
3	2	5	1929.05	1988.04	2080.37	59.31	2.98
4	3	4	1802.00	1852.50	1872.00	33.76	1.82
5							
6							
7	4	3	2634.00	2708.33	2801.00	85.00	3.14
8							
9	5	3	2000.00	2000.00	2000.00	0.00	0.00
10	1	3	1800.00	1870.00	1970.00	88.88	4.75
11	8	3	1660.00	1706.67	1780.00	64.29	3.77
12							
13							
14							
15	6	3	223.00	272.53	371.60	85.79	31.48
16	7	3	1924.00	2017.33	2149.00	117.30	5.81
17							
18							
19							
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	5.00	5.00	5.00	0.00	0.00
2							
3	2	5	76.30	80.15	85.60	3.21	4.00
4	3	4	75.80	77.40	79.30	1.45	1.88
5							
6							
7	4	3	107.00	120.00	132.00	12.53	10.44
8							
9							
10	1	3	41.00	49.33	59.00	9.07	18.39
11	8	3	170.00	193.33	210.00	20.82	10.77
12							
13							
14							
15	6	2	378.20	385.85	393.50	10.82	2.80
16	7	3	228.00	229.67	231.00	1.53	0.67
17							
18							
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	40.00	43.00	45.00	2.65	6.15
2							
3	2	5	487.31	556.56	675.82	69.57	12.50
4	3	4	440.00	459.50	478.00	16.84	3.67
5							
6							
7	4	3	589.00	706.00	767.00	101.36	14.36
8							
9							
10	1	3	350.00	383.33	410.00	30.55	7.97
11	8	3	460.00	476.67	500.00	20.82	4.37
12							
13							
14							
15	6	2	68.00	68.00	68.00	0.00	0.00
16	7	3	579.00	642.67	738.00	84.10	13.09
17							
18							
19							
20							
21							
22							

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
4 - 3	-98.22	-10.18	-10.18	77.86		
4 - 2	-96.16	13.65	13.65	123.45		
2 - 11	-1249.42	-1126.65	-1126.65	-1003.88	***	
2 - 12	-212.72	-89.95	-89.95	32.82		
2 - 1	-149.01	-50.03	-50.03	48.95		
2 - 5	-162.76	-39.99	-39.99	82.78		
2 - 3	-127.58	-23.83	-23.83	79.93		
2 - 4	-123.45	-13.65	-13.65	96.16		

units: mg/kg
optional for all samples
reported by 9 laboratories

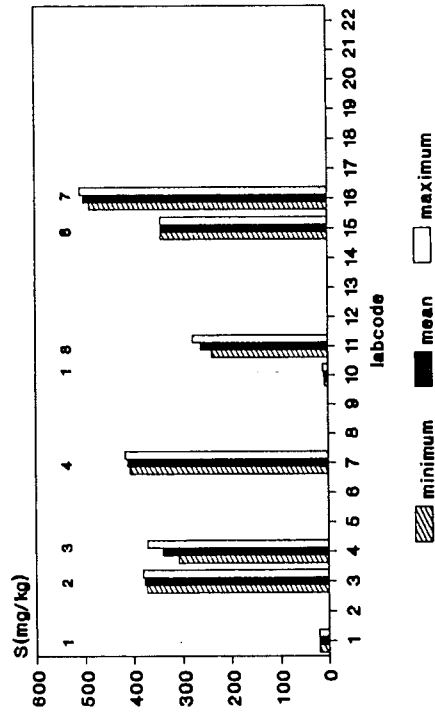
TABLE I : S - Overview of reported data

labcode	samples reported	number of replicates	N method
1	A1 A2 B1 B2	3	N
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8			
9	A2	3	N
10	A1 A2 B1 B2	3	N
11	A1 A2 B1 B2	3	N
12			
13			
14			
15	A1 A2 B1 B2	2	N
16	A1 A2 B1 B2	3	N
17			
18			
19			
20			
21			
22			

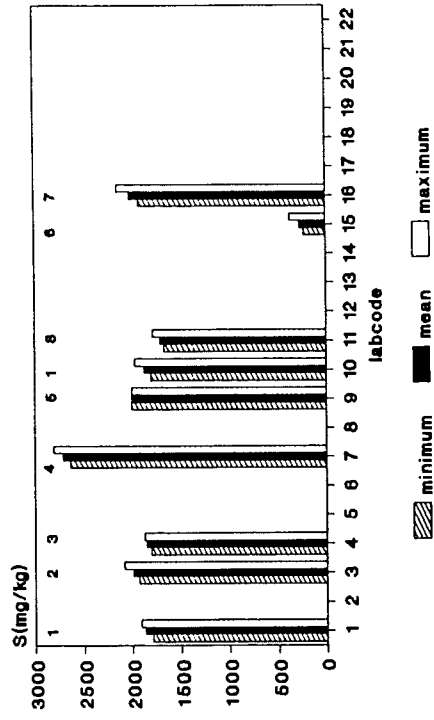
TABLE II : S - Summary method description

method	description
1	LECO
2	digestion, HClO4 + HNO3
3	2M HNO3
4	X-ray fluorescence
5	dry combustion
6	HClO4
7	digestion, HNO3 under pressure
8	unspecified

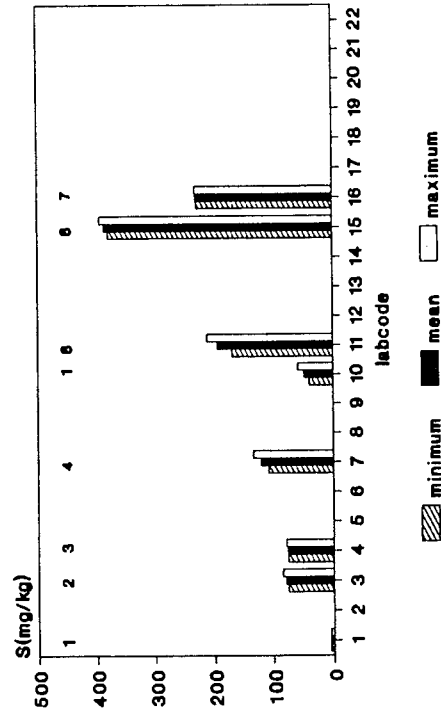
S sample A1
national methods



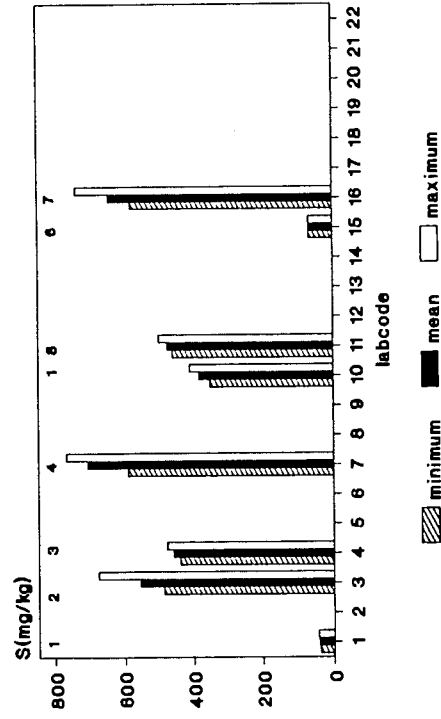
S sample A2
national methods



S sample B1
national methods



S sample B2
national methods



----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 6 7 8

Number of observations in by group = 22

Dependent Variable: S

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	653258.470277	428.39	0.0001
Error	15	3812.250000		
Corrected Total	21	657070.720277		

R-Square C.V. S Mean

0.994198	5.826291	273.62318182

Source	DF	Type I SS	F Value	Pr > F
METH	6	653258.470277	428.39	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	653258.470277	428.39	0.0001

TABLE IV : S - Total mean and normal distribution

sample	method	repl	mean	std	normal	pr < W
A1	1	6	13.83	6.88	0.77	0.03
A1	2	1	376.71			
A1	3	4	338.25	28.49	0.96	0.74
A1	4	3	411.00	5.00	1.00	0.99
A1	6	2	340.50	0.00		
A1	7	6	382.17	131.01	0.77	0.03
A2	1	6	1868.33	70.26	0.93	0.63
A2	2	1	1988.04			
A2	3	4	1852.50	33.76	0.70	0.01
A2	4	3	2708.33	85.00	0.97	0.64
A2	5	3	2000.00	0.00		
A2	6	3	272.53	85.79	0.75	0.00
A2	7	6	1862.00	190.03	0.94	0.64
B1	1	6	27.17	24.95	0.80	0.06
B1	2	1	80.15			
B1	3	4	77.40	1.45	0.98	0.86
B1	4	3	120.00	12.53	1.00	0.87
B1	6	2	385.85	10.82	1.00	1.00
B1	7	6	221.50	23.88	0.85	0.15
B2	1	6	213.17	187.41	0.75	0.02
B2	2	1	556.56			
B2	3	4	459.50	16.84	0.97	0.84
B2	4	3	706.00	101.36	0.77	0.05
B2	6	2	68.00	0.00		
B2	7	6	559.67	106.16	0.90	0.37

Remark:
Labcode 3 reported mean values for 5 replicates.
To calculate the total number of replicates for the method groups, SAS counts the number of reported results.
So, the "repl" values in this table for method 2 are different from the real number of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: S

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 15 MSE= 254.15

Critical Value of Studentized Range= 4.782

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous		Difference		Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit	Difference Between Means	Difference Between Means		
8	- 3	-116.085		-74.917		-33.748	*
8	- 1	211.386		249.500		287.614	*
1	- 7	-525.281		-487.167		-449.052	*
1	- 4	-435.281		-397.167		-359.052	*
1	- 2	-421.098		-362.877		-304.656	*
1	- 6	-370.677		-326.667		-282.656	*
1	- 3	-359.210		-324.417		-289.623	*
1	- 8	-287.614		-249.500		-211.386	*

METH Comparison		Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit			
7	- 4	45.989	134.011	90.000	***	
7	- 2	62.049	186.531	124.290	***	
7	- 6	111.294	209.706	160.500	***	
7	- 3	121.582	203.918	162.750	***	
7	- 8	193.656	281.677	237.667	***	
7	- 1	449.052	525.281	487.167	***	
4	- 7	-134.011	-45.989	-90.000	***	
4	- 2	-27.951	96.531	34.290	***	
4	- 6	21.294	119.706	70.500	***	
4	- 3	31.582	113.918	72.750	***	
4	- 8	103.656	191.677	147.667	***	
4	- 1	359.052	435.281	397.167	***	
2	- 7	-186.531	-62.049	-124.290	***	
2	- 4	-96.531	27.951	-34.290	***	
2	- 6	-29.806	102.226	36.210	***	
2	- 3	-21.804	98.724	38.460	***	
2	- 8	51.136	175.617	113.377	***	
2	- 1	304.656	421.098	362.877	***	
6	- 7	-209.706	-111.294	-160.500	***	
6	- 4	-119.706	-21.294	-70.500	***	
6	- 2	-102.226	29.806	-36.210	***	
6	- 3	-44.431	48.931	2.250	***	
6	- 8	27.961	126.372	77.167	***	
6	- 1	282.656	370.677	326.667	***	
3	- 7	-203.918	-121.582	-162.750	***	
3	- 4	-113.918	-31.582	-72.750	***	
3	- 2	-98.724	21.804	-38.460	***	
3	- 6	-48.931	44.431	-2.250	***	
3	- 8	33.748	116.085	74.917	***	
3	- 1	289.623	359.210	324.417	***	
8	- 7	-281.677	-193.656	-237.667	***	
8	- 4	-191.677	-103.656	-147.667	***	
8	- 2	-175.617	-51.136	-113.377	***	
8	- 6	-126.372	-27.961	-77.167	***	

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 2 3 4 5 6 7 8

Number of observations in by group = 26

Tukey's Studentized Range (HSD) Test for variable: S

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 18 MSE= 5169.758
Critical Value of Studentized Range= 4.824

Comparisons significant at the 0.05 level are indicated by '****'.

Dependent Variable: S				METH Comparison				Simultaneous Lower Confidence Limit				Simultaneous Upper Confidence Limit			
Source	DF	Sum of Squares	F Value	Pr > F											
Model	7	9836676.78585	271.82	0.0001	4	- 7		490.73	691.00	891.27	***	908.60	1003.52	***	***
Error	18	93055.64000			4	- 5		508.06	708.33	908.60	***	1003.52	1013.44	***	***
Corrected Total	25	9929732.42585			4	- 2		437.07	720.29	908.60	***	1013.44	1043.17	***	***
					4	- 1		666.56	840.00	1013.44	***	1043.17	1201.94	***	***
					4	- 3		668.50	855.83	1043.17	***	1201.94	2636.07	***	***
					4	- 8		801.40	1001.67	1201.94	***	2636.07			
					4	- 6		2235.53	2435.80	2636.07	***				
R-Square		C.V.		S Mean											
0.990629		4.001115		1797.0246154	7	- 4		-891.27	-691.00	-490.73	***	217.60			
					7	- 5		-182.94	17.33	217.60	***	312.52			
					7	- 2		-253.93	29.29	312.52	***	322.44			
					7	- 1		-24.44	149.00	322.44	***	352.17			
					7	- 3		-22.50	164.83	352.17	***	510.94			
					7	- 8		110.40	310.67	510.94	***	1945.07			
					7	- 6		1544.53	1744.80	1945.07	***				
					5	- 4		-908.60	-708.33	-508.06	***	182.94			
					5	- 7		-217.60	-17.33	182.94	***	295.18			
					5	- 2		-271.26	11.96	295.18	***	305.11			
					5	- 1		-41.77	131.67	305.11	***	334.84			
					5	- 3		-39.84	147.50	334.84	***	493.60			
					5	- 8		93.06	293.33	493.60	***	1927.74			
					5	- 6		1527.20	1727.47	1927.74	***				
					2	- 4		-1003.52	-720.29	-437.07	***	253.93			
					2	- 7		-312.52	-29.29	253.93	***	271.26			
					2	- 5		-295.18	-11.96	271.26	***	384.64			
					2	- 1		-145.23	119.71	384.64	***	409.77			
					2	- 3		-138.69	135.54	409.77	***	564.60			
					2	- 8		-1.85	281.37	564.60	***	1998.73			
					2	- 6		1432.28	1715.51	1998.73	***				
					1	- 4		-1013.44	-840.00	-666.56	***	24.44			
					1	- 7		-322.44	-149.00	24.44	***	41.77			
					1	- 5		-305.11	-131.67	41.77	***	145.23			
					1	- 2		-384.64	-119.71	145.23	***	174.16			
					1	- 3		-142.49	15.83	174.16	***	335.11			
					1	- 8		-11.77	161.67	335.11	***	1769.24			
					1	- 6		1422.36	1595.80	1769.24	***				

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 6 7 8

Number of observations in by group = 22

Dependent Variable: S

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	249655.390720	141.16	0.0001
Error	15	4421.551667		
Corrected Total	21	254076.942386		

R-Square	C.V.	S Mean
0.982598	12.78895	134.24772727

Source	DF	Type I SS	F Value	Pr > F
METH	6	249655.390720	141.16	0.0001

Source	DF	Type III SS	F Value	Pr > F
METH	6	249655.390720	141.16	0.0001

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
3 - 4	-1043.17	-855.83	-668.50	***
3 - 7	-352.17	-164.83	22.50	
3 - 5	-334.84	-147.50	39.84	
3 - 2	-409.77	-135.54	138.69	
3 - 1	-174.16	-15.83	142.49	
3 - 8	-41.50	145.83	333.17	***
3 - 6	1392.63	1579.97	1767.30	***
8 - 4	-1201.94	-1001.67	-801.40	***
8 - 7	-510.94	-310.67	-110.40	***
8 - 5	-493.60	-293.33	-93.06	***
8 - 2	-564.60	-281.37	1.85	
8 - 1	-335.11	-161.67	11.77	
8 - 3	-333.17	-145.83	41.50	***
8 - 6	1233.86	1434.13	1634.40	***
6 - 4	-2636.07	-2435.80	-2235.53	***
6 - 7	-1945.07	-1744.80	-1544.53	***
6 - 5	-1927.74	-1727.47	-1527.20	***
6 - 2	-1998.73	-1715.51	-1432.28	***
6 - 1	-1769.24	-1595.80	-1422.36	***
6 - 3	-1767.30	-1579.97	-1392.63	***
6 - 8	-1634.40	-1434.13	-1233.86	***

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: S

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 15 MSE= 294.7701
Critical Value of Studentized Range= 4.782

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
3 - 2	-67.652	-2.750	62.152
3 - 1	12.762	50.233	87.704
1 - 6	-406.081	-358.683	-311.286
1 - 7	-243.548	-202.500	-161.452
1 - 8	-207.214	-166.167	-125.119
1 - 4	-133.881	-92.833	-51.786
1 - 2	-115.684	-52.983	9.718
1 - 3	-87.704	-50.233	-12.762

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit
6 - 7	103.191	156.183	209.175
6 - 8	139.525	192.517	245.509
6 - 4	212.858	265.850	318.842
6 - 2	234.604	305.700	376.796
6 - 3	258.177	308.450	358.723
6 - 1	311.286	358.683	406.081
7 - 6	-209.175	-156.183	-103.191
7 - 8	-11.064	36.333	83.731
7 - 4	62.269	109.667	157.064
7 - 2	82.486	149.517	216.547
7 - 3	107.930	152.267	196.603
7 - 1	161.452	202.500	243.548
8 - 6	-245.509	-192.517	-139.525
8 - 7	-83.731	-36.333	11.064
8 - 4	25.936	73.333	120.731
8 - 2	46.153	113.183	180.214
8 - 3	71.597	115.933	160.270
8 - 1	125.119	166.167	207.214
4 - 6	-318.842	-265.850	-212.858
4 - 7	-157.064	-109.667	-62.269
4 - 8	-120.731	-73.333	-25.936
4 - 2	-27.180	39.850	106.880
4 - 3	-1.736	42.600	86.936
4 - 1	51.786	92.833	133.881
2 - 6	-376.796	-305.700	-234.604
2 - 7	-216.547	-149.517	-82.486
2 - 8	-180.214	-113.183	-46.153
2 - 4	-106.880	-39.850	27.180
2 - 3	-62.152	2.750	67.652
2 - 1	-9.718	52.983	115.684
3 - 6	-358.723	-308.450	-258.177
3 - 7	-196.603	-152.267	-107.930
3 - 8	-160.270	-115.933	-71.597
3 - 4	-86.936	-42.600	1.736

SAMPLE=B2

SAMPLE=B2

General Linear Models Procedure

General Linear Models Procedure

Class Level Information

Tukey's Studentized Range (HSD) Test for variable: S

Class	Levels	Values
1	1	1
2	2	2
3	3	3
4	4	4
5	5	5
6	6	6
7	7	7
8	8	8
9	9	9
10	10	10
11	11	11
12	12	12
13	13	13
14	14	14
15	15	15
16	16	16
17	17	17
18	18	18
19	19	19
20	20	20
21	21	21
22	22	22
23	23	23
24	24	24
25	25	25
26	26	26
27	27	27
28	28	28
29	29	29
30	30	30
31	31	31
32	32	32
33	33	33
34	34	34
35	35	35
36	36	36
37	37	37
38	38	38
39	39	39
40	40	40
41	41	41
42	42	42
43	43	43
44	44	44
45	45	45
46	46	46
47	47	47
48	48	48
49	49	49
50	50	50
51	51	51
52	52	52
53	53	53
54	54	54
55	55	55
56	56	56
57	57	57
58	58	58
59	59	59
60	60	60
61	61	61
62	62	62
63	63	63
64	64	64
65	65	65
66	66	66
67	67	67
68	68	68
69	69	69
70	70	70
71	71	71
72	72	72
73	73	73
74	74	74
75	75	75
76	76	76
77	77	77
78	78	78
79	79	79
80	80	80
81	81	81
82	82	82
83	83	83
84	84	84
85	85	85
86	86	86
87	87	87
88	88	88
89	89	89
90	90	90
91	91	91
92	92	92
93	93	93
94	94	94
95	95	95
96	96	96
97	97	97
98	98	98
99	99	99
100	100	100

METH 7 1 2 3 4 6 7 8

Number of observations in by group = 22

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 15 MSE= 14135.28
Critical Value of Studentized Range= 4.782

Comparisons significant at the 0.05 level are indicated by '***'.

Dependent Variable: S				
Source	DF	Sum of Squares	F Value	Pr > F
Model	6	933046.116315	11.00	0.0001
Error	15	212029.166667		
Corrected Total	21	1145075.282982		
	R-Square	C.V.	S Mean	
	0.814834	28.16870	422.07090909	
Source	DF	Type I SS	F Value	Pr > F
METH	6	933046.116315	11.00	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	933046.116315	11.00	0.0001

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
1 - 7	-264.89	63.33	391.55		
4 - 2	-314.73	149.44	613.61		
4 - 8	-98.89	229.33	557.55		
4 - 3	-60.52	246.50	553.52		
4 - 1	208.59	492.83	777.08		
4 - 6	271.04	638.00	1004.96		
7 - 4	-391.55	-63.33	264.89		
7 - 2	-378.07	86.11	550.28		
7 - 8	-162.22	166.00	494.22		
7 - 3	-123.86	183.17	490.19		
7 - 1	145.25	429.50	713.75		
7 - 6	207.70	574.67	941.63		
2 - 4	-613.61	-149.44	314.73		
2 - 7	-550.28	-86.11	378.07		
2 - 8	-384.28	79.89	544.07		
2 - 3	-352.38	97.06	546.50		
2 - 1	-90.80	343.39	777.59		
2 - 6	-3.77	488.56	980.89		
8 - 4	-557.55	-229.33	98.89		
8 - 7	-494.22	-166.00	162.22		
8 - 2	-544.07	-79.89	384.28		
8 - 3	-289.86	17.17	324.19		
8 - 1	-20.75	263.50	547.75		
8 - 6	41.70	408.67	775.63		
3 - 4	-553.52	-246.50	60.52		
3 - 7	-490.19	-183.17	123.86		
3 - 2	-546.50	-97.06	352.38		
3 - 8	-324.19	-17.17	289.86		
3 - 1	-13.15	246.33	505.82		
3 - 6	43.37	391.50	739.63		
1 - 4	-777.08	-492.83	-208.59		
1 - 7	-713.75	-429.50	-145.25		
1 - 2	-777.59	-343.39	90.80		
1 - 8	-547.75	-263.50	20.75		

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
1 - 3	-505.82	-246.33		13.15		
1 - 6	-183.05	145.17		473.39		
6 - 4	-1004.96	-638.00		-271.04		***
6 - 7	-941.63	-574.67		-207.70		***
6 - 2	-980.89	-488.56		3.77		
6 - 8	-775.63	-408.67		-41.70		***
6 - 3	-739.63	-391.50		-43.37		***
6 - 1	-473.39	-145.17		183.05		

units: mg/kg
optional for all samples
reported by 11 laboratories

TABLE I : AI - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1			
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8			
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11			
12			
13			
14	A1 A2 B1 B2	5	N
15	A1 A2 B1 B2	3	N
16	A1 A2 B1 B2	3	R A2:N/R
17			
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20	A2	1 notreported	N
21			
22			

(*) R = aqua regia

TABLE II : AI - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HClO4+HNO3
3	total analysis, 2M HNO3
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO3 under pressure
7	extractant HF
* 8	2N HCl
* 9	minéralisation

* not applicable

TABLE III : A1 - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	16872.37	17430.88	17830.48	371.00	2.13
4	3	4	7321.00	8117.50	9078.00	793.98	9.78
5							
6							
7	4	3	59976.00	60622.00	60996.00	561.77	0.93
8							
9	2	3	13400.00	13700.00	13900.00	264.58	1.93
10	1	3	11100.00	13450.00	14800.00	2042.67	15.19
11							
12							
13							
14	5	5	4181.00	6940.60	8594.00	1643.45	23.68
15	2	4	10000.00	10510.25	11031.00	460.94	4.39
16	1	3	16730.00	16873.33	17060.00	169.21	1.00
17							
18	3	3	9681.46	9741.75	9830.82	78.73	0.81
18	1	3	14843.76	15369.71	15743.94	468.88	3.05
19	1	3		10632.00			
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	20099.36	20850.52	21846.14	569.85	2.73
4	3	4	10609.00	10819.25	11142.00	228.77	2.12
5							
6							
7	4	3	49878.00	50184.00	50592.00	367.77	0.73
8							
9	2	3	16200.00	16633.33	17500.00	750.56	4.51
10	1	3	18000.00	18983.33	20100.00	1056.33	5.57
11							
12							
13							
14	5	5	12708.00	13673.40	14250.00	615.16	4.50
15	2	4	11977.00	14660.25	18109.00	2970.65	20.26
16	1	3	20120.00	20483.33	20970.00	438.22	2.14
17							
18	1	3	17629.54	18197.26	18578.56	501.22	2.75
18	3	3	12626.60	12742.65	12948.36	178.64	1.40
19	1	3		10803.00			
20							
21							
22							

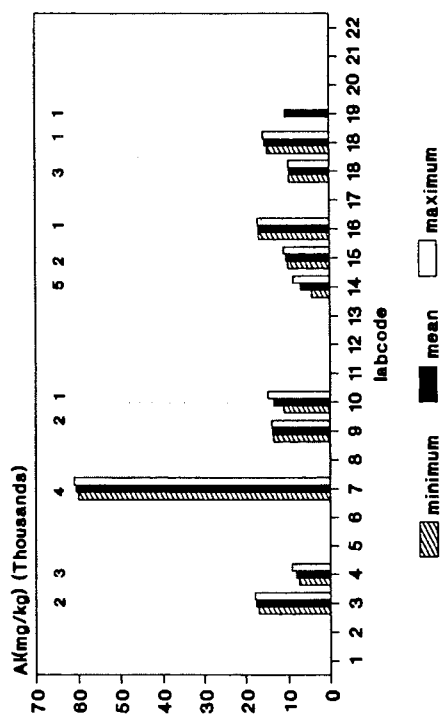
sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	11647.12	12972.18	14253.54	1221.99	9.42
4	3	4	5686.00	5946.75	6191.00	207.24	3.49
5							
6							
7	4	3	14856.00	17339.33	20534.00	2905.07	16.75
8							
9	2	3	9770.00	9770.00	9770.00	0.00	0.00
10	1	3	9300.00	10283.33	11550.00	1151.45	11.20
11							
12							
13							
14	5	5	7058.00	7699.20	8222.00	561.25	7.29
14	A3	5	6886.00	7432.00	7900.00	495.09	6.66
15	2	2	6671.00	6888.50	7106.00	307.59	4.47
16	6	3	13850.00	14396.67	15460.00	921.00	6.40
16	1	3	7590.00	9596.67	10790.00	1748.18	18.22
17							
18	3	3	5632.83	5769.07	5908.93	138.09	2.39
18	1	3	8684.04	8868.47	9044.93	180.58	2.04
19	1	3		10484.00			
20	7	1		670.00			
21							
22							

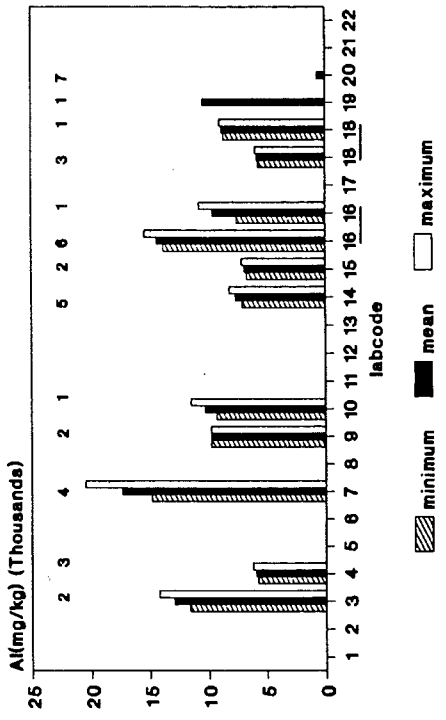
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	2544.09	2762.10	3114.75	201.01	7.28
4	3	4	1908.00	1997.00	2064.00	65.68	3.29
5							
6							
7	4	3	7956.00	8058.00	8160.00	102.00	1.27
8							
9	2	3	2600.00	2800.00	3000.00	200.00	7.14
10	1	3	1850.00	2016.67	2200.00	175.59	8.71
11							
12							
13							
14	5	5	2473.00	2571.20	2607.00	56.18	2.19
15	2	4	1774.10	1791.40	1814.90	18.96	1.06
16	1	3	2500.00	2573.33	2610.00	63.51	2.47
17							
18	1	3	2015.90	2051.66	2118.12	57.61	2.81
18	3	3	1871.19	1911.33	1966.32	49.27	2.58
19	1	3		2000.00			
20							
21							
22							

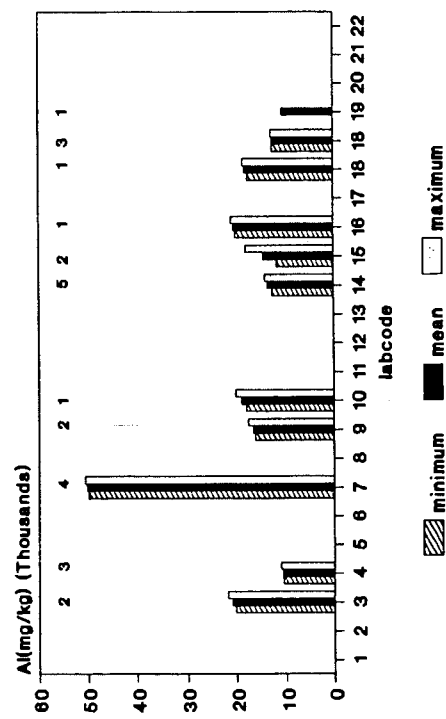
AI sample A1
national methods



AI sample A2
national methods



AI sample B1
national methods



AI sample B2
national methods

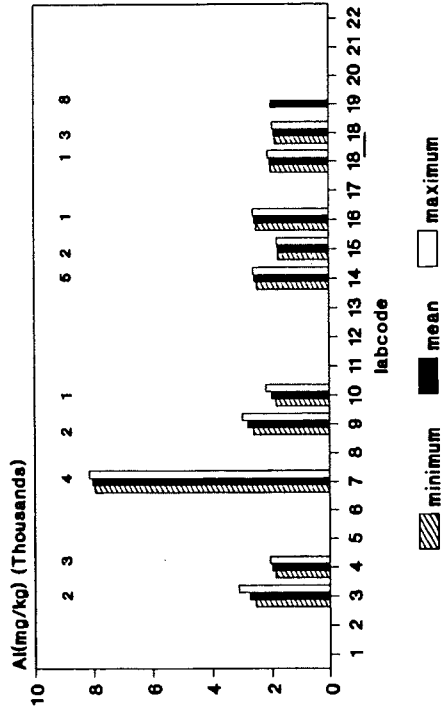


TABLE V : AI Method comparison.

SAMPLE=AI

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 5 1 2 3 4 5

Number of observations in by group = 33

Dependent Variable: AI

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	6854317533.02	441.76	0.0001
Error	28	108610787.01		
Corrected Total	32	6962928320.03		
R-Square C.V. AI Mean				
		12.34338	15955.977273	
Source	DF	Type I SS	F Value	Pr > F
METH	4	6854317533.02	441.76	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	4	6854317533.02	441.76	0.0001

TABLE IV : AI - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	10	14771.110	2249.523	0.847	0.052
A1	2	8	12571.485	2541.274	0.882	0.200
A1	3	7	8813.606	1034.908	0.869	0.190
A1	4	3	60622.000	561.772	0.824	0.174
A1	5	5	6940.600	1643.450	0.832	0.141
A2	1	10	9672.941	1181.509	0.978	0.951
A2	2	6	9343.197	2274.277	0.891	0.318
A2	3	7	5870.601	191.963	0.955	0.790
A2	4	3	17339.333	2905.068	0.955	0.592
A2	5	5	7699.200	561.248	0.794	0.073
A2	6	3	14396.667	920.996	0.764	0.031
A2	7	1	670.000			
A3	5	5	7432.000	495.089	0.815	0.105
B1	1	10	18379.480	2886.623	0.728	0.002
B1	2	8	16173.940	2909.824	0.927	0.491
B1	3	7	11645.564	1045.847	0.801	0.042
B1	4	3	50184.000	367.766	0.942	0.537
B1	5	5	13673.400	615.164	0.910	0.461
B2	1	10	2192.498	279.044	0.850	0.057
B2	2	8	2290.963	544.932	0.788	0.022
B2	3	7	1960.286	71.153	0.949	0.722
B2	4	3	8058.000	102.000	1.000	0.992
B2	5	5	2571.200	56.180	0.723	0.019

Remark:

Labcode 3 and labcode 19 reported mean values for respectively 3 and 5 replicates.

To calculate the total number of replicates for the method-grouped laboratories,

SAS counts the number of reported results.

So, the "repl" values in this table for methods 1 and 2 are different from the real number of replicates.

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Number of observations in by group = 35

Dependent Variable: Al				
Source	DF	Sum of Squares	F Value	Pr > F
Model	6	442620704.084	35.32	0.0001
Error	28	58481760.303		
Corrected Total	34	501102464.388		
R-Square C.V. Al Mean				
		0.883294	15.40939	9378.7657143
Type I SS F Value Pr > F				
Source	DF	Type I SS	F Value	Pr > F
METH	6	442620704.084	35.32	0.0001
Type III SS F Value Pr > F				
Source	DF	Type III SS	F Value	Pr > F
METH	6	442620704.084	35.32	0.0001

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Al

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSE= 3878957
Critical Value of Studentized Range= 4.120

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
4 - 1	42073.6	45850.9	49628.2	***
4 - 2	44165.8	48050.5	51935.3	***
4 - 3	47848.7	51808.4	55768.1	***
4 - 5	49490.8	53681.4	57872.0	***
1 - 4	-49628.2	-45850.9	-42073.6	***
1 - 2	-522.2	2199.6	4921.5	***
1 - 3	3129.7	5957.5	8785.3	***
1 - 5	4687.6	7830.5	10973.4	***
2 - 4	-51935.3	-48050.5	-44165.8	***
2 - 1	-4921.5	-2199.6	522.2	***
2 - 3	788.1	3757.9	6727.7	***
2 - 5	2359.6	5630.9	8902.1	***
3 - 4	-55768.1	-51808.4	-47848.7	***
3 - 1	-8785.3	-5957.5	-3129.7	***
3 - 2	-6727.7	-3757.9	-788.1	***
3 - 5	-1486.9	1873.0	5232.9	***
5 - 4	-57872.0	-53681.4	-49490.8	***
5 - 1	-10973.4	-7830.5	-4687.6	***
5 - 2	-8902.1	-5630.9	-2359.6	***
5 - 3	-5232.9	-1873.0	1486.9	***

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Al

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSE= 2088634
 Critical Value of Studentized Range= 4.486

Comparisons significant at the 0.05 level are indicated by '***'.

General Linear Models Procedure			
METH Comparison	Simultaneous		Simultaneous Upper Confidence Limit
	Lower Confidence Limit	Difference Between Means	
3 - 5	-4513.0	-1828.6	855.8
3 - 7	299.7	5200.6	10101.5
7 - 4	-21963.0	-16669.3	-11375.7
7 - 6	-19020.3	-13726.7	-8433.0
7 - 1	-13811.1	-9002.9	-4194.8
7 - 2	-13624.9	-8673.2	-3721.5
7 - 5	-12051.2	-7029.2	-2007.2
7 - 3	-10101.5	-5200.6	-299.7

General Linear Models Procedure			
METH Comparison	Simultaneous		Simultaneous Upper Confidence Limit
	Lower Confidence Limit	Difference Between Means	
4 - 6	-800.5	2942.7	6685.8
4 - 1	4648.6	7666.4	10684.2
4 - 2	4754.5	7996.1	11237.8
4 - 5	6292.2	9640.1	12988.1
4 - 3	8305.2	11468.7	14632.3
4 - 7	11375.7	16669.3	21963.0
6 - 4	-6685.8	-2942.7	800.5
6 - 1	1705.9	4723.7	7741.6
6 - 2	1811.8	5053.5	8295.1
6 - 5	3349.5	6697.5	10045.4
6 - 3	5362.5	8526.1	11689.6
6 - 7	8433.0	13726.7	19020.3
1 - 4	-10684.2	-7666.4	-4648.6
1 - 6	-7741.6	-4723.7	-1705.9
1 - 2	-2037.6	329.7	2697.1
1 - 5	-537.2	1973.7	4484.7
1 - 3	1543.1	3802.3	6061.6
1 - 7	4194.8	9002.9	13811.1
2 - 4	-11237.8	-7996.1	-4754.5
2 - 6	-8295.1	-5053.5	-1811.8
2 - 1	-2697.1	-329.7	2037.6
2 - 5	-1132.0	1644.0	4420.0
2 - 3	922.1	3472.6	6023.1
2 - 7	3721.5	8673.2	13624.9
5 - 4	-12988.1	-9640.1	-6292.2
5 - 6	-10045.4	-6697.5	-3349.5
5 - 1	-4484.7	-1973.7	537.2
5 - 2	-4420.0	-1644.0	1132.0
5 - 3	-855.8	1828.6	4513.0
5 - 7	2007.2	7029.2	12051.2
3 - 4	-14632.3	-11468.7	-8305.2
3 - 6	-11689.6	-8526.1	-5362.5
3 - 1	-6061.6	-3802.3	-1543.1
3 - 2	-6023.1	-3472.6	-922.1

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Al

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSF= 5093210
Critical Value of Studentized Range= 4.120

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison		Simultaneous		Difference		Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit		
4	- 1	27476	31805		36133	***	
4	- 2	29559	34010		38462	***	
4	- 5	31709	36511		41312	***	
4	- 3	34003	38540		43078	***	
1	- 4	-36133	-31805		-27476	***	
1	- 2	-913	2206		5324	***	
1	- 5	1105	4706		8307	***	
1	- 3	3496	6736		9976	***	
2	- 4	-38462	-34010		-29559	***	
2	- 1	-5324	-2206		913		
2	- 5	-1248	2501		6249		
2	- 3	1127	4530		7933	***	
5	- 4	-41312	-36511		-31709	***	
5	- 1	-8307	-4706		-1105	***	
5	- 2	-6249	-2501		1248		
5	- 3	-1820	2030		5880		
3	- 4	-43078	-38540		-34003	***	
3	- 1	-9976	-6736		-3496	***	
3	- 2	-7933	-4530		-1127	***	
3	- 5	-5880	-2030		1820		

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Al

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 28 MSE= 101544.9
Critical Value of Studentized Range= 4.120

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison			Simultaneous Lower		Difference Between Means	Simultaneous Upper	
			Confidence Limit	Limit		Confidence Limit	Limit
1	-	5	4808.8		5486.8	6164.8	***
1	-	2	5138.5		5767.0	6395.6	***
1	-	1	5254.3		5865.5	6476.7	***
1	-	3	5457.0		6097.7	6738.4	***
5	-	4	-6164.8		-5486.8	-4808.8	***
5	-	2	-249.0		280.2	809.5	
5	-	1	-129.8		378.7	887.2	
5	-	3	67.3		610.9	1154.5	***
2	-	4	-6395.6		-5767.0	-5138.5	***
2	-	5	-809.5		-280.2	249.0	
2	-	1	-341.9		98.5	538.9	
2	-	3	-149.8		330.7	811.2	
1	-	4	-6476.7		-5865.5	-5254.3	***
1	-	5	-887.2		-378.7	129.8	
1	-	2	-538.9		-98.5	341.9	
1	-	3	-225.3		232.2	689.7	
3	-	4	-6738.4		-6097.7	-5457.0	***
3	-	5	-1154.5		-610.9	-67.3	***
3	-	2	-811.2		-330.7	149.8	
3	-	1	-689.7		-232.2	225.3	

28. Fe

units: mg/kg
optional for all samples
reported by 14 laboratories

TABLE I : Fe - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11			
12			
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	A1 B2:4 A2:2 B1:3	N
16	A1 A2 B1 B2	3	R A2: N/R
17			
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20	A2	1 not reported	N
21			
22	A2	3	R

(*) R = aqua regia

TABLE II : Fe - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HNO ₃ + HClO ₄
3	total analysis, 2M HNO ₃
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO ₃ under pressure
7	extractant HF
8	2N HCl
9	minéralisation

TABLE III : Fe - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	2499.00	2499.00	2499.00	0.00	0.00
2							
3	2	5	23558.50	23645.62	23798.64	87.68	0.37
4	3	4	13240.00	14552.50	16256.00	1412.35	9.71
5							
6							
7	4	3	28356.00	28492.00	28560.00	117.78	0.41
8	9	3	26000.00	26000.00	26000.00	0.00	0.00
9	2	3	20600.00	20800.00	21100.00	264.58	1.27
10	1	3	17520.00	17708.33	18015.00	267.88	1.51
11							
12							
13							
14	5	5	4304.00	9202.00	13385.00	3234.94	35.16
15	2	4	16490.00	17612.00	18775.00	1127.70	6.40
16	1	3	22770.00	22980.00	23170.00	200.75	0.87
17							
18	1	3	19710.40	20150.67	20828.00	595.32	2.95
18	3	3	16357.60	16493.07	16560.80	117.32	0.71
19	1	3		21650.00			
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	4419.00	4499.00	4550.00	70.15	1.56
2							
3	2	5	8673.60	9588.93	10518.78	707.58	7.38
4	3	4	6342.00	6648.00	6974.00	258.27	3.89
5							
6							
7	4	3	9271.00	10716.33	12611.00	1714.74	16.00
8	9	3	8212.00	8339.67	8407.00	110.62	1.33
9	2	3	7170.00	7493.33	7820.00	325.01	4.34
10	1	3	7480.00	7813.33	8010.00	290.23	3.72
11							
12							
13							
14	5	5	7554.00	8210.60	8800.00	602.54	7.34
14	A3	5	6886.00	7708.60	8173.00	595.39	7.72
15	2	2	6570.90	6626.60	6682.30	78.77	1.19
16	6	3	8070.00	8403.33	9030.00	543.08	6.46
16	1	3	6940.00	8866.67	9860.00	1668.81	18.82
17							
18	1	3	8804.70	9022.10	9239.50	217.40	2.41
18	3	3	7065.50	7137.97	7174.20	62.76	0.88
19	1	3		8800.00			
20	7	1		5720.00			
21							
22	1	3	16530.00	16683.67	16818.00	144.97	0.87

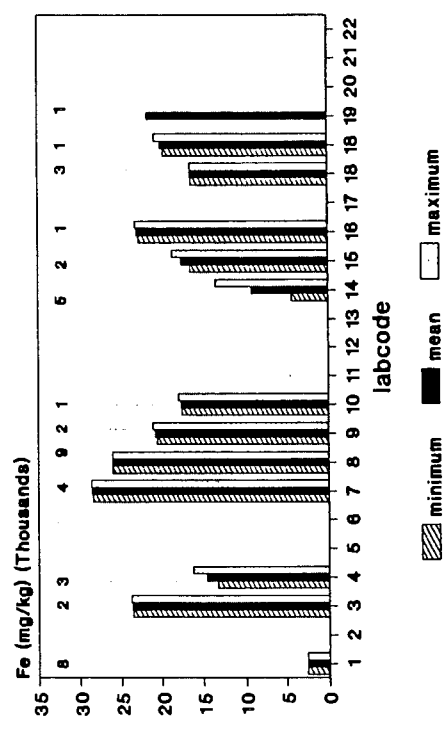
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	1870.00	1870.00	1870.00	0.00	0.00
2							
3	2	5	25279.14	26033.08	28619.91	1290.62	4.96
4	3	4	15063.00	15468.50	15857.00	328.61	2.12
5							
6							
7	4	3	28356.00	28492.00	28662.00	155.81	0.55
8	9	3	25500.00	25602.00	25704.00	102.00	0.40
9	2	3	21700.00	22100.00	22500.00	400.00	1.81
10	1	3	18120.00	18758.33	19300.00	595.91	3.18
11							
12							
13							
14	5	5	17797.00	19157.40	19891.00	832.54	4.35
15	2	3	20567.00	21499.00	22200.00	840.65	3.91
16	1	3	24690.00	25146.67	25670.00	493.39	1.96
17							
18	1	3	22837.50	22905.17	22939.00	58.60	0.26
18	3	3	17559.50	17728.67	18067.00	293.01	1.65
19	1	3		18830.00			
20							
21							
22							

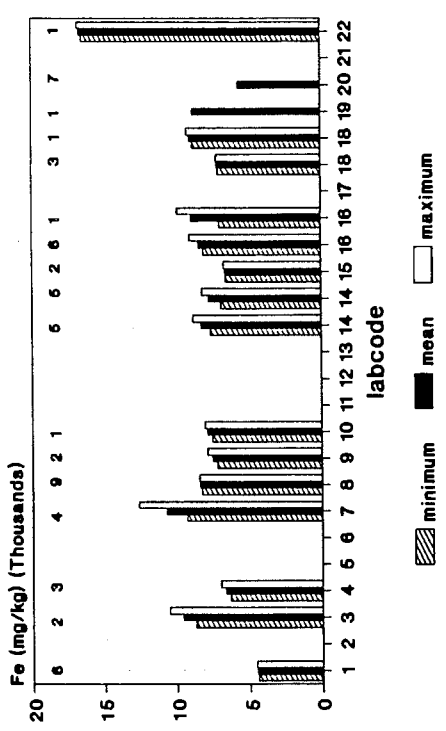
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	906.00	957.33	1044.00	75.48	7.88
2							
3	2	5	1694.40	1784.31	1995.97	115.04	6.45
4	3	4	1397.00	1454.50	1498.00	42.26	2.91
5							
6							
7	4	3	2754.00	2922.00	3156.00	208.97	7.15
8	9	3	1932.00	1966.00	2034.00	58.89	3.00
9	2	3	1700.00	1800.00	1900.00	100.00	5.56
10	1	3	1320.00	1340.00	1360.00	20.00	1.49
11							
12							
13							
14	5	5	1500.00	1574.20	1626.00	48.66	3.09
15	2	4	1141.90	1166.90	1223.50	37.99	3.26
16	1	3	1750.00	1776.67	1820.00	37.86	2.13
17							
18	1	3	1418.82	1453.90	1520.02	57.30	3.94
18	3	3	1238.69	1299.07	1346.97	55.21	4.25
19	1	3		1493.00			
20							
21							
22							

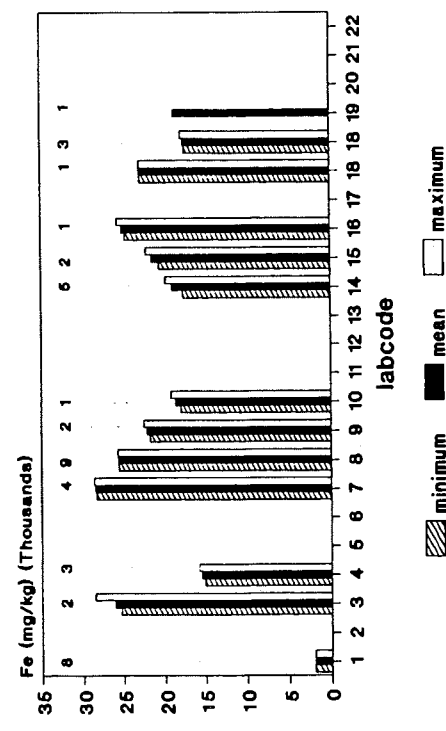
Fe sample A1
national/reference method



Fe sample A2
national/reference method



Fe sample B1
national/reference method



Fe sample B2
national/reference method

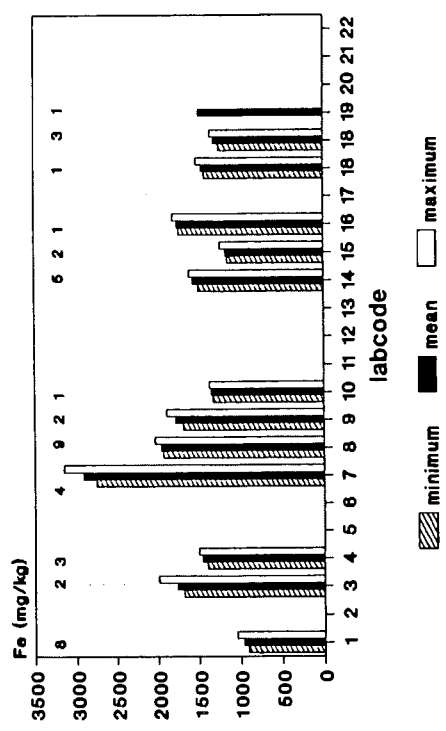


TABLE V : Fe Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 8 9

Number of observations in by group = 39

Dependent Variable: Fe

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1748803889.42	67.02	0.0001
Error	32	139177014.05		
Corrected Total	38	1887980903.47		
R-Square		C.V.		Fe Mean
	0.926283	11.86784		17572.636410

Source	DF	Type I SS	F Value	Pr > F
METH	6	1748803889.42	67.02	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	1748803889.42	67.02	0.0001

TABLE IV : Fe - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	10	20416.70	2220.70	0.90	0.23
A1	2	8	19561.70	2403.54	0.95	0.71
A1	3	7	15384.17	1441.49	0.79	0.03
A1	4	3	28492.00	117.78	0.75	0.00
A1	5	5	9202.00	3234.94	0.91	0.48
A1	8	3	2499.00	0.00		
A1	9	3	26000.00			
A2	1	10	10458.25	3647.67	0.74	0.00
A2	2	6	7553.69	1103.59	0.86	0.18
A2	3	7	6857.99	321.33	0.89	0.26
A2	4	3	10716.33	1714.74	0.95	0.56
A2	5	5	8210.60	602.54	0.82	0.11
A2	6	3	8403.33	543.08	0.78	0.07
A2	7	1	5720.00			
A2	8	3	4499.00	70.15	0.87	0.30
A2	9	3	8339.67	110.62	0.78	0.06
A3	5	5	7708.60	595.39	0.81	0.09
B1	1	10	21926.05	2884.55	0.88	0.12
B1	2	7	22404.30	1714.54	0.79	0.03
B1	3	7	16437.14	1241.83	0.85	0.12
B1	4	3	28492.00	155.81	0.96	0.64
B1	5	5	19157.40	832.54	0.88	0.31
B1	8	3	1870.00	0.00		
B1	9	3	25602.00	102.00	1.00	0.99
B2	1	10	1520.47	188.24	0.87	0.08
B2	2	8	1481.49	341.48	0.79	0.02
B2	3	7	1387.89	93.87	0.95	0.72
B2	4	3	2922.00	208.97	0.93	0.47
B2	5	5	1574.20	48.66	0.95	0.71
B2	8	3	957.33	75.48	0.84	0.20
B2	9	3	1966.00	58.89	0.75	0.00

Remark:
Labcode 3 and labcode 19 reported mean values and the number of replicates, respectively 5 and 3.
To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.
So, the "repl" values in this table for methods 1 and 2 are different from the real number of replicates.

SAMPLE=Al

SAMPLE=Al

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 32 MSE= 4349282
Critical Value of Studentized Range= 4.445

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	5	3	5	3	5	3
5	-10020.5	-6182.2	-23501.0	-2343.9	1915.8	11490.2
5	-10020.5	-6182.2	-23501.0	-2343.9	1915.8	11490.2
8	-31345.2	-25993.0	-23501.0	-20640.8	-28853.2	-18148.8
8	-31345.2	-25993.0	-23501.0	-20640.8	-28853.2	-18148.8
8	-2232.8	-17917.7	-17917.7	-13602.6	-2232.8	-13602.6
8	-2232.8	-17917.7	-17917.7	-13602.6	-2232.8	-13602.6
8	-21500.6	-17062.7	-17062.7	-12624.9	-21500.6	-12624.9
8	-21500.6	-17062.7	-17062.7	-12624.9	-21500.6	-12624.9
8	-17408.6	-12885.2	-12885.2	-8361.7	-17408.6	-8361.7
8	-17408.6	-12885.2	-12885.2	-8361.7	-17408.6	-8361.7
5	-11490.2	-6703.0	-6703.0	-1915.8	-11490.2	-1915.8

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	4	9	4	9	4	9
4	-2860.2	2492.0	2492.0	7844.2	3760.2	12390.4
4	-2860.2	2492.0	2492.0	7844.2	3760.2	12390.4
4	4492.4	8930.3	8930.3	13368.1	4492.4	13368.1
4	4492.4	8930.3	8930.3	13368.1	4492.4	13368.1
4	8584.4	13107.8	13107.8	17631.3	8584.4	17631.3
4	8584.4	13107.8	13107.8	17631.3	8584.4	17631.3
4	14502.8	19290.0	19290.0	24077.2	14502.8	24077.2
4	14502.8	19290.0	19290.0	24077.2	14502.8	24077.2
4	20640.8	25993.0	25993.0	31345.2	20640.8	31345.2
4	20640.8	25993.0	25993.0	31345.2	20640.8	31345.2
9	-7844.2	-2492.0	-2492.0	2860.2	-1268.2	9898.4
9	-7844.2	-2492.0	-2492.0	2860.2	-1268.2	9898.4
9	1268.2	5583.3	5583.3	10876.1	1268.2	10876.1
9	1268.2	5583.3	5583.3	10876.1	1268.2	10876.1
9	2000.4	6438.3	6438.3	15139.3	2000.4	15139.3
9	2000.4	6438.3	6438.3	15139.3	2000.4	15139.3
9	6092.4	10615.8	10615.8	21585.2	6092.4	21585.2
9	6092.4	10615.8	10615.8	21585.2	6092.4	21585.2
9	12010.8	16798.0	16798.0	28853.2	12010.8	28853.2
9	12010.8	16798.0	16798.0	28853.2	12010.8	28853.2
9	18148.8	23501.0	23501.0	3760.2	18148.8	3760.2
9	18148.8	23501.0	23501.0	3760.2	18148.8	3760.2
1	-12390.4	-8075.3	-8075.3	-3760.2	-9898.4	-1268.2
1	-12390.4	-8075.3	-8075.3	-3760.2	-9898.4	-1268.2
1	-9898.4	-5583.3	-5583.3	10876.1	-2254.4	3964.4
1	-9898.4	-5583.3	-5583.3	10876.1	-2254.4	3964.4
1	-2254.4	855.0	855.0	8262.9	1802.1	14805.1
1	-2254.4	855.0	855.0	8262.9	1802.1	14805.1
1	1802.1	5032.5	5032.5	14805.1	7624.3	2232.8
1	1802.1	5032.5	5032.5	14805.1	7624.3	2232.8
1	7624.3	11214.7	11214.7	2232.8	13602.6	17917.7
1	7624.3	11214.7	11214.7	2232.8	13602.6	17917.7
1	13602.6	17917.7	17917.7	2232.8	13602.6	17917.7
1	13602.6	17917.7	17917.7	2232.8	13602.6	17917.7
2	-13368.1	-8930.3	-8930.3	-4492.4	-10876.1	-2000.4
2	-13368.1	-8930.3	-8930.3	-4492.4	-10876.1	-2000.4
2	-10876.1	-6438.3	-6438.3	2254.4	-3964.4	7570.1
2	-10876.1	-6438.3	-6438.3	2254.4	-3964.4	7570.1
2	-3964.4	-855.0	-855.0	8262.9	784.9	14096.7
2	-3964.4	-855.0	-855.0	8262.9	784.9	14096.7
2	784.9	4177.5	4177.5	14096.7	6622.7	21500.6
2	784.9	4177.5	4177.5	14096.7	6622.7	21500.6
2	6622.7	10359.7	10359.7	21500.6	12624.9	17062.7
2	6622.7	10359.7	10359.7	21500.6	12624.9	17062.7
2	12624.9	17062.7	17062.7	21500.6	12624.9	17062.7
2	12624.9	17062.7	17062.7	21500.6	12624.9	17062.7
3	-17631.3	-13107.8	-13107.8	-8584.4	-15139.3	-6092.4
3	-17631.3	-13107.8	-13107.8	-8584.4	-15139.3	-6092.4
3	-15139.3	-10615.8	-10615.8	17631.3	-8262.9	-1802.1
3	-15139.3	-10615.8	-10615.8	17631.3	-8262.9	-1802.1
3	-8262.9	-5032.5	-5032.5	24077.2	-7570.1	10020.5
3	-8262.9	-5032.5	-5032.5	24077.2	-7570.1	10020.5
3	-7570.1	-4177.5	-4177.5	10020.5	2343.9	17408.6
3	-7570.1	-4177.5	-4177.5	10020.5	2343.9	17408.6
3	2343.9	6182.2	6182.2	17408.6	8361.7	14502.8
3	2343.9	6182.2	6182.2	17408.6	8361.7	14502.8
3	8361.7	12885.2	12885.2	24077.2	-14805.1	-11214.7
3	8361.7	12885.2	12885.2	24077.2	-14805.1	-11214.7
4	-24077.2	-19290.0	-19290.0	-14502.8	-24077.2	-14502.8
4	-24077.2	-19290.0	-19290.0	-14502.8	-24077.2	-14502.8
5	-21585.2	-16798.0	-16798.0	-7624.3	-21585.2	-16798.0
5	-21585.2	-16798.0	-16798.0	-7624.3	-21585.2	-16798.0
5	-14805.1	-11214.7	-11214.7	-6622.7	-14805.1	-11214.7
5	-14805.1	-11214.7	-11214.7	-6622.7	-14805.1	-11214.7
5	-14096.7	-10359.7	-10359.7	24077.2	-14096.7	-10359.7
5	-14096.7	-10359.7	-10359.7	24077.2	-14096.7	-10359.7

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 4980916
Critical Value of Studentized Range= 4.671

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous			Simultaneous		
		Lower Limit	Confidence Limit	Difference Between Means	Upper Limit	Confidence Limit	Upper Limit
4	- 1	-4463.3		258.1	4979.5		
4	- 6	-3705.6		2313.0	8331.6		
4	- 9	-3641.9		2376.7	8395.3		
4	- 5	-2877.5		2505.7	7888.9		
4	- 2	-2049.6		3162.6	8374.9		
4	- 3	-1228.3		3858.3	8945.0		
4	- 7	-3515.3		4996.3	13507.9		***
4	- 8	198.7		6217.3	12235.9		***
1	- 4	-4979.5		-258.1	4463.3		
1	- 6	-2666.5		2054.9	6776.3		
1	- 9	-2602.8		2118.6	6840.0		
1	- 5	-1631.4		2247.7	6126.7		
1	- 2	-733.5		2904.6	6542.6		***
1	- 3	144.6		3600.3	7056.0		***
1	- 7	-2911.3		4738.3	12387.8		***
1	- 8	1237.9		5959.3	10680.6		***
6	- 4	-8331.6		-2313.0	3705.6		
6	- 1	-6776.3		-2054.9	2666.5		
6	- 9	-5954.9		63.7	6082.3		
6	- 5	-5190.5		192.7	5575.9		
6	- 2	-4362.6		849.6	6061.9		
6	- 3	-3541.3		1545.3	6632.0		
6	- 7	-5828.3		2683.3	11194.9		
6	- 8	-2114.3		3904.3	9922.9		
9	- 4	-8395.3		-2376.7	3641.9		
9	- 1	-6840.0		-2118.6	2602.8		
9	- 6	-6082.3		-63.7	5954.9		
9	- 5	-5254.1		129.1	5512.3		
9	- 2	-4426.3		786.0	5998.3		
9	- 3	-3605.0		1481.7	6568.3		
9	- 7	-5891.9		2619.7	11131.3		
9	- 8	-2177.9		3840.7	9859.3		
5	- 4	-7888.9		-2505.7	2877.5		
5	- 1	-6126.7		-2247.7	1631.4		
5	- 6	-5575.9		-192.7	5190.5		

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	9	1 2 3 4 5 6 7 8 9

Number of observations in by group = 44

Dependent Variable: Fe					Fe Mean	
Source	DF	Sum of Squares	F Value	Pr > F		
Model	8	145014014.442	3.64	0.0035		
Error	35	174332068.463				
Corrected Total	43	319346082.905				
R-Square					C.V.	
					Fe Mean	
					8453.0302273	
Type I SS					F Value	
Source	DF	Type I SS	F Value	Pr > F		
METH	8	145014014.442	3.64	0.0035		
Type III SS					F Value	
Source	DF	Type III SS	F Value	Pr > F		
METH	8	145014014.442	3.64	0.0035		

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 8 9

Number of observations in by group = 38

Dependent Variable: Fe

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1465845327.83	72.39	0.0001
Error	31	104618522.81		
Corrected Total	37	1570463850.64		

R-Square C.V. Fe Mean

0.933384 9.248220 19863.936316

Source	DF	Type I SS	F Value	Pr > F
METH	6	1465845327.83	72.39	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	1465845327.83	72.39	0.0001

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit

5	- 9	-5512.3	-129.1	5254.1		
5	- 2	-3806.6	656.9	5120.4		
5	- 3	-2963.6	1352.6	5668.8		
5	- 7	-5584.2	2490.6	10565.4		
5	- 8	-1671.6	3711.6	9094.8		
2	- 4	-8374.9	-3162.6	2049.6		
2	- 1	-6542.6	-2904.6	733.5		
2	- 6	-6061.9	-849.6	4362.6		
2	- 9	-5998.3	-786.0	4426.3		
2	- 5	-5120.4	-656.9	3806.6		
2	- 3	-3405.3	695.7	4796.7		
2	- 7	-6128.2	1833.7	9795.6		
2	- 8	-2157.6	3054.7	8267.0		
3	- 4	-8945.0	-3858.3	1228.3		
3	- 1	-7056.0	-3600.3	-144.6		***
3	- 6	-6632.0	-1545.3	3541.3		
3	- 9	-6568.3	-1481.7	3605.0		
3	- 5	-5668.8	-1352.6	2963.6		
3	- 2	-4796.7	-695.7	3405.3		
3	- 7	-6742.2	1138.0	9018.2		
3	- 8	-2727.7	2359.0	7445.6		
7	- 4	-13507.9	-4996.3	3515.3		
7	- 1	-12387.8	-4738.3	2911.3		
7	- 6	-11194.9	-2683.3	5828.3		
7	- 9	-11131.3	-2619.7	5891.9		
7	- 5	-10565.4	-2490.6	5584.2		
7	- 2	-9795.6	-1833.7	6128.2		
7	- 3	-9018.2	-1138.0	6742.2		
7	- 8	-7290.6	1221.0	9732.6		
8	- 4	-12235.9	-6217.3	-198.7		***
8	- 1	-10680.6	-5959.3	-1237.9		***
8	- 6	-9922.9	-3904.3	2114.3		
8	- 9	-9859.3	-3840.7	2177.9		
8	- 5	-9094.8	-3711.6	1671.6		
8	- 2	-8267.0	-3054.7	2157.6		
8	- 3	-7445.6	-2359.0	2727.7		
8	- 7	-9732.6	-1221.0	7290.6		

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Fe

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 31 MSE= 3374791

Critical Value of Studentized Range= 4.454

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 9	-1834.4	2890.0	7614.4	-6108.3	-2720.3	667.8
4 - 2	2094.8	6087.7	10080.6	10574.3	14567.1	18560.0
4 - 1	2757.0	6566.0	10374.9	-31346.4	-26622.0	-21897.6
4 - 5	5109.0	9334.6	13560.2	-28456.4	-23732.0	-19007.6
4 - 3	8062.0	12054.9	16047.7	-24527.2	-20534.3	-16541.4
4 - 8	21897.6	26622.0	31346.4	-23865.0	-20056.0	-16247.1
9 - 4	-7614.4	-2890.0	1834.4	-21513.0	-17287.4	-13061.8
9 - 2	-795.2	3197.7	7190.6	-18560.0	-14567.1	-10574.3
9 - 1	-133.0	3676.0	7484.9			
9 - 5	2219.0	6444.6	10670.2			
9 - 3	5172.0	9164.9	13157.7			
9 - 8	19007.6	23732.0	28456.4			
2 - 4	-10080.6	-6087.7	-2094.8			
2 - 9	-7190.6	-3197.7	795.2			
2 - 1	-2373.2	478.2	3329.7			
2 - 5	-141.2	3246.9	6635.0			
2 - 3	2874.3	5967.2	9060.0			
2 - 8	16541.4	20534.3	24527.2			
1 - 4	-10374.9	-6566.0	-2757.0			
1 - 9	-7484.9	-3676.0	133.0			
1 - 2	-3329.7	-478.2	2373.2			
1 - 5	-400.6	2768.6	5937.9			
1 - 3	2637.4	5488.9	8340.4			
1 - 8	16247.1	20056.0	23865.0			
5 - 4	-13560.2	-9334.6	-5109.0			
5 - 9	-10670.2	-6444.6	-2219.0			
5 - 2	-6635.0	-3246.9	141.2			
5 - 1	-5937.9	-2768.6	400.6			
5 - 3	-667.8	2720.3	6108.3			
5 - 8	13061.8	17287.4	21513.0			
3 - 4	-16047.7	-12054.9	-8062.0			
3 - 9	-13157.7	-9164.9	-5172.0			
3 - 2	-9060.0	-5967.2	-2874.3			
3 - 1	-8340.4	-5488.9	-2637.4			

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
3 - 2	-421.88	-93.60			234.68	
3 - 8	-7.16	430.55			868.26	
8 - 4	-2482.57	-1964.67			-1446.76	***
8 - 9	-1526.57	-1008.67			-490.76	***
8 - 5	-1080.09	-616.87			-153.64	***
8 - 1	-980.69	-563.14			-145.59	***
8 - 2	-953.58	-524.16			-94.73	***
8 - 3	-868.26	-430.55			7.16	

29. Zn

units: mg/kg
optional for all samples
reported by 13 laboratories

TABLE I : Zn - Overview of reported data

labcode	samples reported			number of replicates	N/R method (*)
1	A1	A2	B1 B2	3	N/R
2					
3	A1	A2	B1 B2	5	N
4	A1	A2	B1 B2	4	N
5					
6					
7	A1	A2	B1 B2	3	N
8	A1	A2	B1 B2	3	N
9	A1	A2	B1 B2	3	N
10	A1	A2	B1 B2	3	R
11					
12					
13					
14	A1	A2	A3 B1 B2	5	N
15	A1	A2	B1 B2	4 A2:2	N
16	A1	A2	B1 B2	3	N/R
17					
18	A1	A2	B1 B2	3	N/R
19	A1	A2	B1 B2	3	R
20					
21					
22			A2	3	R

(*) R = aqua regia

TABLE II : Zn - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HNO3 + HClO4
3	total analysis, 2M HNO3
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO3 under pressure
7	extractant HF
8	2N HCl
9	minéralisation

TABLE IIIa : Zn - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	20.00	20.33	21.00	0.58	2.84
2							
3	2	5	64.93	65.87	66.50	0.59	0.90
4	3	4	47.30	52.15	57.70	5.05	9.67
5							
6							
7	4	3	80.60	82.60	83.60	1.73	2.10
8	9	3	54.00	54.04	54.06	0.04	0.06
9	2	3	67.00	67.33	68.00	0.58	0.86
10							
11							
12							
13							
14	5	5	21.60	33.60	41.40	7.28	21.65
15	2	4	51.35	55.92	58.98	3.31	5.91
16							
17							
18	3	3	54.56	55.41	56.49	0.99	1.78
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	139.00	139.00	139.00	0.00	0.00
2							
3	2	5	145.03	149.48	153.48	3.60	2.41
4	3	4	139.30	142.00	143.10	1.81	1.27
5							
6							
7	4	3	154.00	165.33	175.00	10.60	6.41
8	9	3	1.10	77.63	116.40	66.28	85.38
9	2	3	142.00	142.33	143.00	0.58	0.41
10							
11							
12							
13							
14	5	5	118.00	121.60	125.00	2.51	2.06
14	A3 5	5	118.00	119.40	121.00	1.34	1.12
15	2	2	125.20	127.15	129.10	2.76	2.17
16	6	3	118.00	126.67	132.00	7.57	5.98
17							
18	3	3	130.44	134.79	138.05	3.92	2.91
19							
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	7.00	7.67	9.00	1.16	15.06
2							
3	2	5	49.15	49.90	50.72	0.64	1.28
4	3	4	37.90	38.73	40.40	1.13	2.92
5							
6							
7	4	3	53.00	55.07	56.10	1.79	3.25
8	9	3	42.80	42.81	42.84	0.02	0.05
9	2	3	47.00	47.67	48.00	0.58	1.21
10							
11							
12							
13							
14	5	5	33.30	34.88	35.80	0.98	2.80
15	2	4	35.87	41.84	49.60	5.72	13.88
16							
17							
18	3	3	38.27	38.98	39.59	0.67	1.71
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	8	3	25.00	25.67	27.00	1.16	4.50
2							
3	2	5	20.45	22.88	27.27	2.50	10.93
4	3	4	22.60	23.48	24.10	0.63	2.70
5							
6							
7	4	3	26.50	31.27	33.70	4.13	13.20
8	9	3	20.00	20.57	21.30	0.67	3.24
9	2	3	22.00	22.00	22.00	0.00	0.00
10							
11							
12							
13							
14	5	5	19.60	20.20	21.00	0.52	2.55
15	2	4	19.08	19.85	20.60	0.72	3.61
16							
17							
18	3	3	18.01	19.56	20.44	1.35	6.90
19							
20							
21							
22							

TABLE IIIB : Zn - Descriptive statistics

sample A1 - aqua regia (R)

labcode	repl	minimum	mean	maximum	std	cv
1	3	32.00	32.00	32.00	0.00	0.00
2						
3						
4						
5						
6						
7						
8						
9						
10	3	61.50	63.00	64.00	1.32	2.10
11						
12						
13						
14						
15						
16	3	65.00	66.67	68.00	1.53	2.29
17						
18	3	66.95	67.63	68.36	0.72	1.06
19	3		63.00			
20						
21						
22						

sample A2 - aqua regia (R)

labcode	repl	minimum	mean	maximum	std	cv
1	3	57.00	57.67	58.00	0.58	1.00
2						
3						
4						
5						
6						
7						
8						
9						
10	3	130.00	131.67	134.00	2.08	1.58
11						
12						
13						
14						
15						
16	3	140.00	143.33	146.00	3.06	2.13
17						
18	3	142.40	146.02	148.92	3.32	2.27
19	3		148.00			
20						
21						
22	3	177.00	181.67	185.00	4.16	2.29

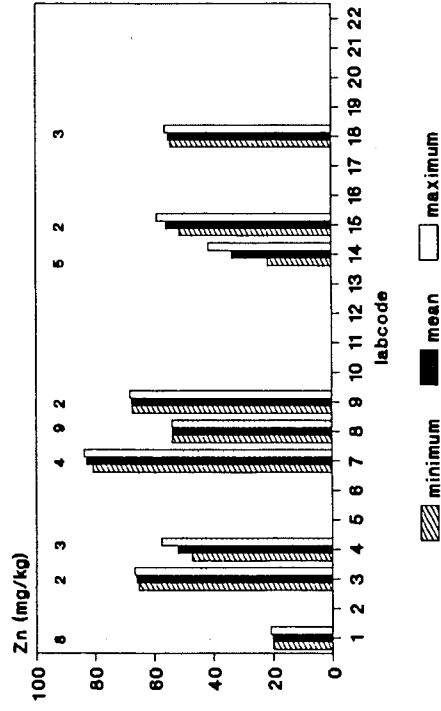
sample B1 - aqua regia (R)

labcode	repl	minimum	mean	maximum	std	cv
1	3	22.00	22.00	22.00	0.00	0.00
2						
3						
4						
5						
6						
7						
8						
9						
10	3	40.50	41.67	42.50	1.04	2.50
11						
12						
13						
14						
15						
16	3	47.00	47.67	48.00	0.58	1.21
17						
18	3	43.75	45.47	46.59	1.51	3.33
19	3		40.00			
20						
21						
22						

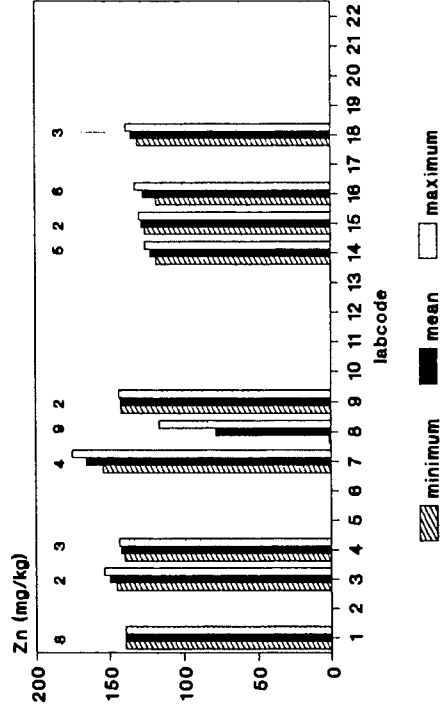
sample B2 - aqua regia (R)

labcode	repl	minimum	mean	maximum	std	cv
1	3	10.00	10.00	10.00	0.00	0.00
2						
3						
4						
5						
6						
7						
8						
9						
10	3	18.50	19.83	21.00	1.26	6.34
11						
12						
13						
14						
15						
16	3	22.00	27.33	36.00	7.57	27.70
17						
18	3	20.14	20.81	21.96	1.00	4.80
19	3		21.00			
20						
21						
22						

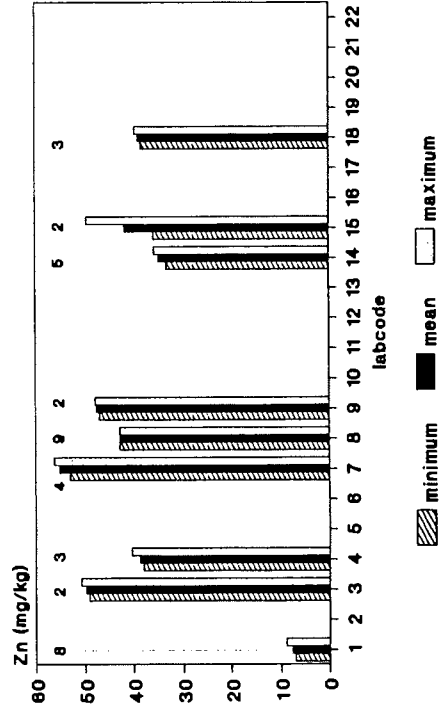
Zn sample A1
national methods



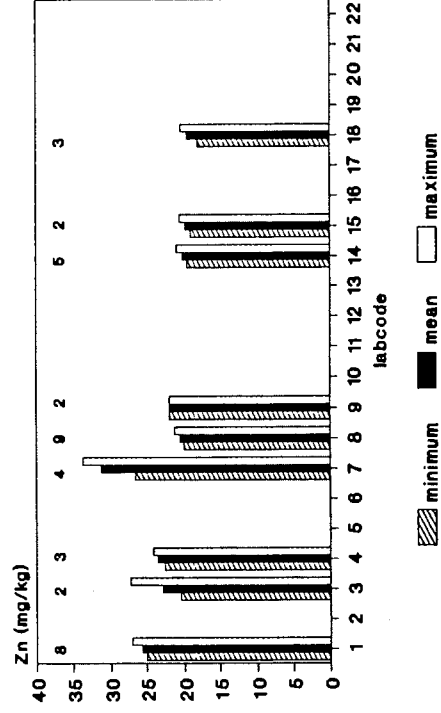
Zn sample A2
national methods



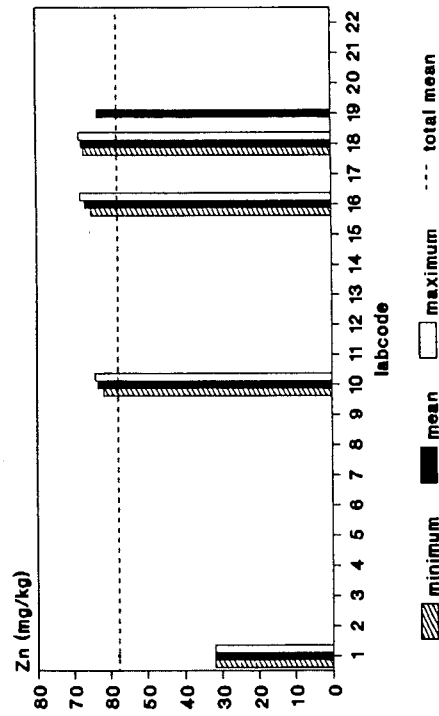
Zn sample B1
national methods



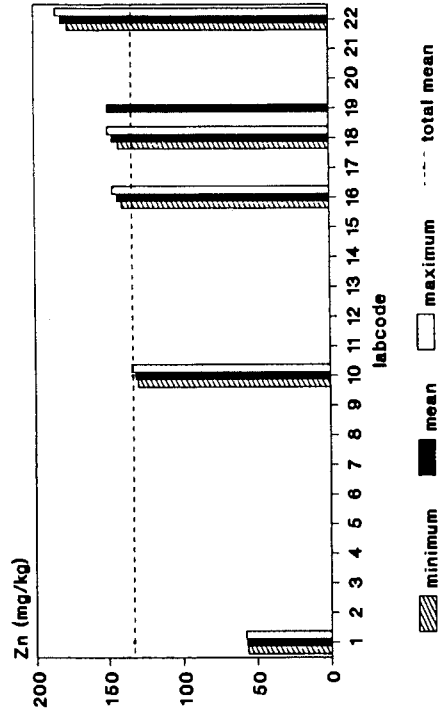
Zn sample B2
national methods



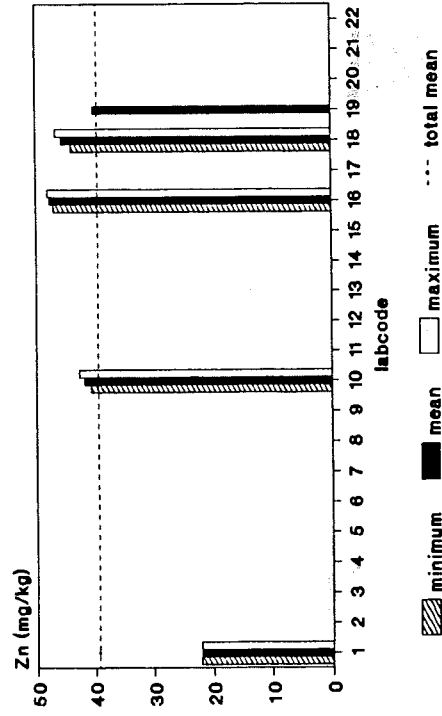
Zn sample A1
reference method



Zn sample A2
reference method



Zn sample B1
reference method



Zn sample B2
reference method

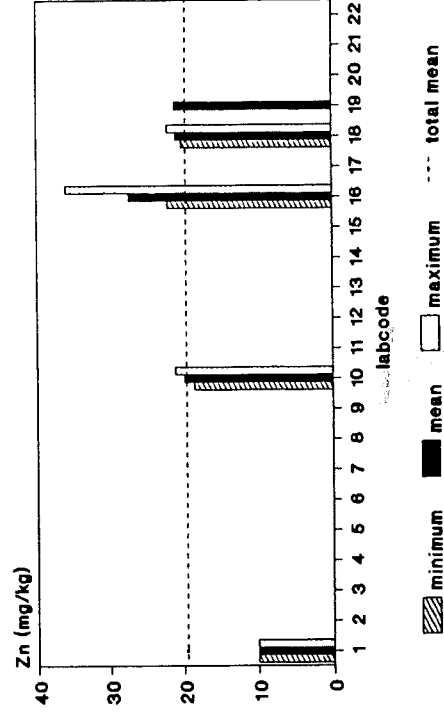


TABLE V : Zn Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 8 9

Number of observations in by group = 42

Dependent Variable: ZN

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	8559.96662972	15.44	0.0001
Error	35	3234.21821790		
Corrected Total	41	11794.18484762		
R-Square				
		C.V.	ZN Mean	
		0.725779	17.89493	53.71809524
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	6	8559.96662972	15.44	0.0001
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	6	8559.96662972	15.44	0.0001

TABLE IV : Zn - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	57.76	14.83	0.65	0.00
A1	2	8	61.44	6.32	0.88	0.19
A1	3	7	53.55	4.01	0.84	0.09
A1	4	3	82.60	1.73	0.75	0.00
A1	5	5	33.60	7.28	0.86	0.22
A1	8	3	20.33	0.58	0.75	0.00
A1	9	3	54.04	0.04	0.75	0.00
A2	1	16	133.13	41.07	0.82	0.00
A2	2	6	138.46	9.28	0.89	0.29
A2	3	7	138.91	4.65	0.88	0.21
A2	4	3	165.33	10.60	0.98	0.74
A2	5	5	121.60	2.51	0.95	0.75
A2	6	3	126.67	7.57	0.86	0.25
A2	8	3	139.00	0.00		
A2	9	3	77.63	66.28	0.76	0.01
A3	5	5	119.40	1.34	0.85	0.20
B1	1	13	39.26	10.19	0.75	0.00
B1	2	8	45.03	5.13	0.86	0.13
B1	3	7	38.83	0.90	0.88	0.24
B1	4	3	55.07	1.79	0.75	0.00
B1	5	5	34.88	0.98	0.90	0.42
B1	8	3	7.67	1.16	0.75	0.00
B1	9	3	42.81	0.02	0.75	0.00
B2	1	13	19.61	6.97	0.85	0.02
B2	2	8	21.03	1.38	0.91	0.38
B2	3	7	21.80	2.28	0.89	0.30
B2	4	3	31.27	4.13	0.76	0.02
B2	5	5	20.20	0.52	0.95	0.74
B2	8	3	25.67	1.16	0.75	0.00
B2	9	3	20.57	0.67	0.95	0.58

Remark:

Labcode 19 reported mean values for 3 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for method 1 are different from the real number
of replicates.

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
5 - 3	-37.541	-19.946	-2.350	-37.541	-2.350
5 - 8	-8.679	13.267	35.212	-8.679	35.212
8 - 4	-86.802	-62.267	-37.731	-86.802	-37.731
8 - 2	-61.452	-41.108	-20.764	-61.452	-20.764
8 - 1	-56.675	-37.427	-18.180	-56.675	-18.180
8 - 9	-58.242	-33.707	-9.171	-58.242	-9.171
8 - 3	-53.949	-33.212	-12.476	-53.949	-12.476
8 - 5	-35.212	-13.267	8.679	-35.212	8.679

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: ZN

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 92.40623
Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
4 - 2	0.815	21.159	21.159	41.503	***
4 - 1	5.592	24.839	24.839	44.087	***
4 - 9	4.024	28.560	28.560	53.096	***
4 - 3	8.318	29.054	29.054	49.791	***
4 - 5	27.055	49.000	49.000	70.945	***
4 - 8	37.731	62.267	62.267	86.802	***
2 - 4	-41.503	-21.159	-21.159	-0.815	***
2 - 1	-9.823	3.680	3.680	17.184	***
2 - 9	-12.943	7.401	7.401	27.745	***
2 - 3	-7.657	7.896	7.896	23.448	***
2 - 5	10.710	27.841	27.841	44.972	***
2 - 8	20.764	41.108	41.108	61.452	***
1 - 4	-44.087	-24.839	-24.839	-5.592	***
1 - 2	-17.184	-3.680	-3.680	9.823	***
1 - 9	-15.527	3.721	3.721	22.968	***
1 - 3	-9.873	4.215	4.215	18.303	***
1 - 5	8.347	24.161	24.161	39.974	***
1 - 8	18.180	37.427	37.427	56.675	***
9 - 4	-53.096	-28.560	-28.560	-4.024	***
9 - 2	-27.745	-7.401	-7.401	12.943	***
9 - 1	-22.968	-3.721	-3.721	15.527	***
9 - 3	-20.242	0.494	0.494	21.231	***
9 - 5	-1.505	20.440	20.440	42.385	***
9 - 8	9.171	33.707	33.707	58.242	***
3 - 4	-49.791	-29.054	-29.054	-8.318	***
3 - 2	-23.448	-7.896	-7.896	7.657	***
3 - 1	-18.303	-4.215	-4.215	9.873	***
3 - 9	-21.231	-0.494	-0.494	20.242	***
3 - 5	2.350	19.946	19.946	37.541	***
3 - 8	12.476	33.212	33.212	53.949	***
5 - 4	-70.945	-49.000	-49.000	-27.055	***
5 - 2	-44.972	-27.841	-27.841	-10.710	***
5 - 1	-39.974	-24.161	-24.161	-8.347	***
5 - 9	-42.385	-20.440	-20.440	1.505	***

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: ZN

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 38 MSE= 921.496
Critical Value of Studentized Range= 4.533

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous		Difference		Simultaneous	
		Confidence	Lower Limit	Between Means	Upper Confidence Limit		
4	- 8	-53.12	26.33	105.78			
4	- 3	-40.72	26.42	93.57			
4	- 2	-41.94	26.87	95.68			
4	- 1	-29.02	32.20	93.43			
4	- 6	-40.78	38.67	118.12			
4	- 5	-27.33	43.73	114.80			
4	- 9	8.25	87.70	167.15		***	
8	- 4	-105.78	-26.33	53.12			
8	- 3	-67.06	0.09	67.24			
8	- 2	-68.27	0.54	69.34			
8	- 1	-55.35	5.87	67.09			
8	- 6	-67.12	12.33	91.78			
8	- 5	-53.66	17.40	88.46			
8	- 9	-18.08	61.37	140.82			
3	- 4	-93.57	-26.42	40.72			
3	- 8	-67.24	-0.09	67.06			
3	- 2	-53.69	0.45	54.58			
3	- 1	-38.32	5.78	49.88			
3	- 6	-54.90	12.24	79.39			
3	- 5	-39.67	17.31	74.29			
3	- 9	-5.87	61.28	128.42			
2	- 4	-95.68	-26.87	41.94			
2	- 8	-69.34	-0.54	68.27			
2	- 3	-54.58	-0.45	53.69			
2	- 1	-41.25	5.33	51.92			
2	- 6	-57.01	11.80	80.60			
2	- 5	-42.06	16.86	75.79			
2	- 9	-7.98	60.83	129.64			
1	- 4	-93.43	-32.20	29.02			
1	- 8	-67.09	-5.87	55.35			
1	- 3	-49.88	-5.78	38.32			
1	- 2	-51.92	-5.33	41.25			
1	- 6	-54.76	6.46	67.68			
1	- 5	-38.33	11.53	61.38			
1	- 9	-5.73	55.50	116.72			

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 8 9

Number of observations in by group = 42

Dependent Variable: ZN

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	4139.87147605	16.67	0.0001
Error	35	1448.93033585		
Corrected Total	41	5588.80181190		
R-Square C.V. ZN Mean				
		0.740744	16.54271	38.89404762

Source	DF	Type I SS	F Value	Pr > F
METH	6	4139.87147605	16.67	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	4139.87147605	16.67	0.0001

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
6 - 4	-118.12	-38.67	40.78	
6 - 8	-91.78	-12.33	67.12	
6 - 3	-79.39	-12.24	54.90	
6 - 2	-80.60	-11.80	57.01	
6 - 1	-67.68	-6.46	54.76	
6 - 5	-66.00	5.07	76.13	
6 - 9	-30.42	49.03	128.48	
5 - 4	-114.80	-43.73	27.33	
5 - 8	-88.46	-17.40	53.66	
5 - 3	-74.29	-17.31	39.67	
5 - 2	-75.79	-16.86	42.06	
5 - 1	-61.38	-11.53	38.33	
5 - 5	-76.13	-5.07	66.00	
5 - 9	-27.10	43.97	115.03	
9 - 4	-167.15	-87.70	-8.25	***
9 - 8	-140.82	-61.37	18.08	
9 - 3	-128.42	-61.28	5.87	
9 - 2	-129.64	-60.83	7.98	
9 - 1	-116.72	-55.50	5.73	
9 - 6	-128.48	-49.03	30.42	
9 - 5	-115.03	-43.97	27.10	

Zn Method comparison

Zn Method comparison

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: ZN

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 41.39801
 Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
5	- 3	-15.731		-3.954		7.823	***
5	- 8	12.525		27.213		41.902	***
8	- 4	-63.822		-47.400		-30.978	***
8	- 2	-50.981		-37.365		-23.748	***
8	- 9	-51.569		-35.147		-18.724	***
8	- 1	-44.479		-31.596		-18.714	***
8	- 3	-45.047		-31.168		-17.288	***
8	- 5	-41.902		-27.213		-12.525	***

METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
4	- 2	-3.581		10.035		23.652	
4	- 9	-4.169		12.253		28.676	***
4	- 1	2.921		15.804		28.686	***
4	- 3	2.353		16.232		30.112	***
4	- 5	5.498		20.187		34.875	***
4	- 8	30.978		47.400		63.822	***
2	- 4	-23.652		-10.035		3.581	
2	- 9	-11.399		2.218		15.835	
2	- 1	-3.270		5.768		14.806	
2	- 3	-4.213		6.197		16.607	
2	- 5	-1.315		10.151		21.618	***
2	- 8	23.748		37.365		50.981	***
9	- 4	-28.676		-12.253		4.169	
9	- 2	-15.835		-2.218		11.399	
9	- 1	-9.333		3.550		16.433	
9	- 3	-9.900		3.979		17.859	
9	- 5	-6.755		7.933		22.622	***
9	- 8	18.724		35.147		51.569	***
1	- 4	-28.686		-15.804		-2.921	***
1	- 2	-14.806		-5.768		3.270	
1	- 9	-16.433		-3.550		9.333	
1	- 3	-9.000		0.429		9.858	
1	- 5	-6.201		4.383		14.967	***
1	- 8	18.714		31.596		44.479	***
3	- 4	-30.112		-16.232		-2.353	***
3	- 2	-16.607		-6.197		4.213	
3	- 9	-17.859		-3.979		9.900	
3	- 1	-9.858		-0.429		9.000	
3	- 5	-7.823		3.954		15.731	***
3	- 8	17.288		31.168		45.047	***
5	- 4	-34.875		-20.187		-5.498	***
5	- 2	-21.618		-10.151		1.315	
5	- 9	-22.622		-7.933		6.755	
5	- 1	-14.967		-4.383		6.201	

SAMPLE=B2

General Linear Models Procedure

Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 8 9

Number of observations in by group = 42

Dependent Variable: ZN

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	397.13996960	3.48	0.0084
Error	35	665.43272802		
Corrected Total	41	1062.57269762		

R-Square C.V. ZN Mean

0.373753 20.14025 21.64976190

Source	DF	Type I SS	F Value	Pr > F
METH	6	397.13996960	3.48	0.0084

Source	DF	Type III SS	F Value	Pr > F
METH	6	397.13996960	3.48	0.0084

Tukey's Studentized Range (HSD) Test for variable: ZN

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 19.01236
Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '****'.

		Simultaneous		Simultaneous	
		Lower	Difference	Upper	
		Confidence	Between	Confidence	
		Limit	Means	Limit	
METH	Comparison				
4	- 8	-5.529	5.600	16.729	***
4	- 3	0.062	9.468	18.874	***
4	- 2	1.006	10.234	19.462	***
4	- 9	-0.429	10.700	21.829	***
4	- 5	1.112	11.067	21.021	***
4	- 1	2.925	11.656	20.386	***
8	- 4	-16.729	-5.600	5.529	
8	- 3	-5.538	3.868	13.274	
8	- 2	-4.594	4.634	13.862	
8	- 9	-6.029	5.100	16.229	
8	- 5	-4.488	5.467	15.421	
8	- 1	-2.675	6.056	14.786	
3	- 4	-18.874	-9.468	-0.062	***
3	- 8	-13.274	-3.868	5.538	
3	- 2	-6.288	0.766	7.821	
3	- 9	-8.174	1.232	10.638	
3	- 5	-6.383	1.599	9.580	
3	- 1	-4.202	2.188	8.578	
2	- 4	-19.462	-10.234	-1.006	***
2	- 8	-13.862	-4.634	4.594	
2	- 3	-7.821	-0.766	6.288	
2	- 9	-8.762	0.466	9.694	
2	- 5	-6.938	0.832	8.603	
2	- 1	-4.703	1.422	7.547	
9	- 4	-21.829	-10.700	0.429	
9	- 8	-16.229	-5.100	6.029	
9	- 3	-10.638	-1.232	8.174	
9	- 2	-9.694	-0.466	8.762	
9	- 5	-9.588	0.367	10.321	
9	- 1	-7.775	0.956	9.686	
5	- 4	-21.021	-11.067	-1.112	***
5	- 8	-15.421	-5.467	4.488	
5	- 3	-9.580	-1.599	6.383	
5	- 2	-8.603	-0.832	6.938	

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
5 - 9	-10.321	-0.367	9.588			
5 - 1	-6.584	0.589	7.762			
1 - 4	-20.386	-11.656	-2.925			***
1 - 8	-14.786	-6.056	2.675			
1 - 3	-8.578	-2.188	4.202			
1 - 2	-7.547	-1.422	4.703			
1 - 9	-9.686	-0.956	7.775			
1 - 5	-7.762	-0.589	6.584			

30. Cu

units: mg/kg
optional for all samples
reported by 13 laboratories

TABLE I : Cu - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N/R
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11			
12			
13			
14	A1 A2 A3 B1 B2	4	N
15	A1 A2 B1 B2	A1 B1:4 A2:2 B2:3	N
16	A1 A2 B1 B2	3	R A2: N/R
17			
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20			
21	A2	3	R
22			

(*) R = aqua regia

TABLE II : Cu - Summary method descriptions

method	description
1	aqua regia
2	HNO3 + HClO4
3	2M HNO3
4	AFNOR X31-151
5	dry ashing
6	digestion, HNO3 under pressure
7	2N HCl
8	minéralisation

TABLE III : Cu - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	10.00	10.00	10.00	0.00	0.00
1	7	3	14.00	14.00	14.00	0.00	0.00
2							
3	2	5	22.28	22.56	22.98	0.25	1.11
4	3	4	18.40	20.43	22.90	2.10	10.27
5							
6							
7	4	3	23.40	23.47	23.50	0.06	0.25
8	8	3	32.45	33.29	34.81	1.32	3.97
9	2	3	26.00	26.00	26.00	0.00	0.00
10	1	3	22.50	23.00	23.50	0.50	2.17
11							
12							
13							
14	5	5	4.03	6.32	7.96	1.42	22.50
15	2	4	19.63	21.00	22.24	1.20	5.70
16	1	3	14.40	14.57	14.70	0.15	1.05
17							
18	1	3	28.35	28.55	28.75	0.20	0.70
18	3	3	20.93	21.30	21.74	0.41	1.92
19	1	3		26.00			
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	5.00	5.00	5.00	0.00	0.00
1	7	3	7.00	7.00	7.00	0.00	0.00
2							
3	2	5	13.89	14.17	14.64	0.29	2.05
4	3	4	11.40	11.60	11.70	0.14	1.22
5							
6							
7	4	3	14.30	15.23	16.60	1.21	7.94
8	8	3	16.47	17.25	17.67	0.67	3.91
9	2	3	14.00	14.00	14.00	0.00	0.00
10	1	3	12.50	12.50	12.50	0.00	0.00
11							
12							
13							
14	5	5	8.29	8.64	9.54	0.52	5.97
14	A3	5	8.71	8.81	8.94	0.10	1.10
15	2	2	12.87	13.84	14.81	1.37	9.91
16	1	3	9.00	9.40	9.60	0.35	3.69
16	6	3	9.70	10.30	11.10	0.72	7.00
17							
18	1	3	17.17	17.90	18.48	0.67	3.73
18	3	3	11.30	11.52	11.63	0.19	1.65
19	1	3		15.00			
20							
21							
22	1	3	22.00	22.67	24.00	1.16	5.09

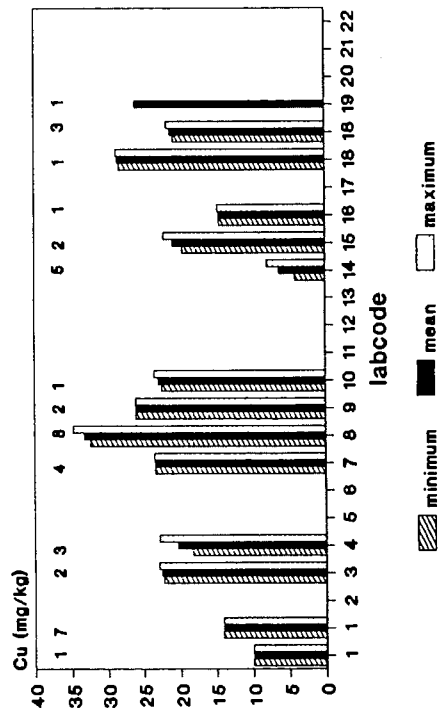
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	4.00	4.00	4.00	0.00	0.00
1	7	3	3.00	3.00	3.00	0.00	0.00
2							
3	2	5	9.94	10.41	10.90	0.36	3.46
4	3	4	7.61	7.86	8.28	0.29	3.73
5							
6							
7	4	3	13.00	13.60	13.90	0.52	3.82
8	8	3	14.65	15.21	15.50	0.49	3.19
9	2	3	13.00	13.33	14.00	0.58	4.33
10	1	3	10.00	10.33	10.50	0.29	2.79
11							
12							
13							
14	5	5	5.11	6.31	7.18	0.78	12.36
15	2	4	9.75	10.85	11.94	0.98	8.99
16	1	3	4.20	4.40	4.60	0.20	4.55
17							
18	1	3	14.01	14.72	15.53	0.77	5.20
18	3	3	10.05	10.22	10.45	0.21	2.04
19	1	3		11.00			
20							
21							
22							

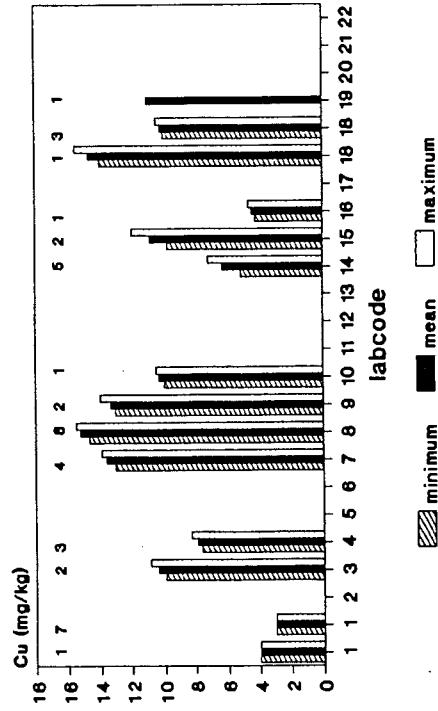
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	1.00	1.00	1.00	0.00	0.00
1	7	3	2.00	2.00	2.00	0.00	0.00
2							
3	2	5	3.38	4.06	4.97	0.55	13.55
4	3	4	1.56	1.59	1.60	0.02	1.26
5							
6							
7	4	3	6.20	6.57	7.30	0.64	9.67
8	8	3	6.58	6.78	7.01	0.22	3.18
9	2	3	4.00	4.33	5.00	0.58	13.32
10	1	3	3.00	3.33	3.50	0.29	8.66
11							
12							
13							
14	5	5	1.86	1.99	2.08	0.09	4.37
15	2	3	3.32	3.62	3.93	0.31	8.43
16	1	3	1.50	1.63	1.80	0.15	9.35
17							
18	3	3	2.73	2.97	3.24	0.26	8.66
18	1	3	6.27	6.34	6.48	0.12	1.91
19	1	3		3.80			
20							
21							
22							

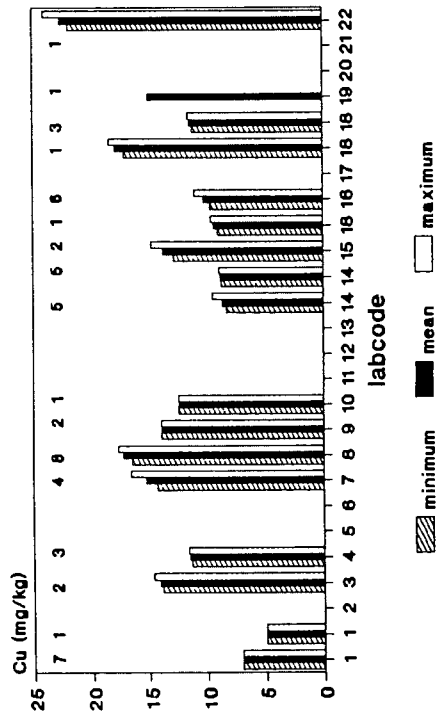
Cu sample A1
national/reference methods



Cu sample B1
national/reference methods



Cu sample A2
national/reference methods



Cu sample B2
national/reference methods

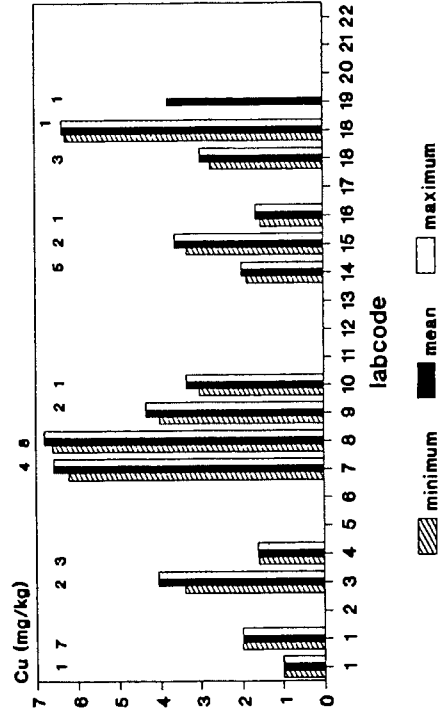


TABLE V : Cu Method comparison

----- SAMPLE=A1 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 42

Dependent Variable: Cu

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1688.28514038	13.26	0.0001
Error	35	742.90022391		
Corrected Total	41	2431.18536429		

R-Square C.V. Cu Mean

0.694429 23.35854 19.72357143

Source	DF	Type I SS	F Value	Pr > F
METH	6	1688.28514038	13.26	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	1688.28514038	13.26	0.0001

TABLE IV : Cu - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	19.57	7.47	0.87	0.05
A1	2	8	23.07	2.60	0.86	0.13
A1	3	7	20.80	1.57	0.92	0.50
A1	4	3	23.47	0.06	0.75	0.00
A1	5	5	6.32	1.42	0.87	0.27
A1	7	3	14.00	0.00		
A1	8	3	33.29	1.32	0.80	0.11
A2	1	16	13.59	6.25	0.94	0.35
A2	2	6	13.98	0.63	0.86	0.17
A2	3	7	11.57	0.16	0.82	0.07
A2	4	3	15.23	1.21	0.90	0.40
A2	5	5	8.64	0.52	0.74	0.03
A2	6	3	10.30	0.72	0.94	0.54
A2	7	3	7.00	0.00		
A2	8	3	17.25	0.67	0.79	0.10
A3	5	5	8.81	0.10	0.92	0.56
B1	1	13	8.57	4.52	0.84	0.02
B1	2	8	11.72	1.52	0.94	0.63
B1	3	7	8.87	1.29	0.80	0.04
B1	4	3	13.60	0.52	0.75	0.00
B1	5	5	6.31	0.78	0.95	0.72
B1	7	3	3.00	0.00		
B1	8	3	15.21	0.49	0.77	0.04
B2	1	13	3.13	2.08	0.85	0.03
B2	2	7	3.99	0.52	0.88	0.24
B2	3	7	2.18	0.75	0.76	0.02
B2	4	3	6.57	0.64	0.75	0.00
B2	5	5	1.99	0.09	0.93	0.60
B2	7	3	2.00	0.00		
B2	8	3	6.78	0.22	0.99	0.82

Remark:
Labcode 3 and labcode 19 reported mean values and the number of replicates, respectively 5 and 3.
To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.
So, the "repl" values in this table for methods 1 and 2 are different from the real number of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cu

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 21.22572

Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '***'.

General Linear Models Procedure			General Linear Models Procedure		
METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
7 - 1	-14.790	-5.565	3.659	7 - 1	-14.790
7 - 5	-2.840	7.678	18.196	7 - 5	-2.840
5 - 8	-37.482	-26.965	-16.447	5 - 8	-37.482
5 - 4	-27.662	-17.145	-6.627	5 - 4	-27.662
5 - 2	-24.960	-16.749	-8.539	5 - 2	-24.960
5 - 3	-22.911	-14.478	-6.045	5 - 3	-22.911
5 - 1	-20.822	-13.243	-5.665	5 - 1	-20.822
5 - 7	-18.196	-7.678	2.840	5 - 7	-18.196

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
8 - 4	-1.939	9.820	21.579	8 - 4	-1.939
8 - 2	0.465	10.215	19.966	8 - 2	0.465
8 - 3	2.548	12.487	22.425	8 - 3	2.548
8 - 1	4.497	13.721	22.946	8 - 1	4.497
8 - 7	7.527	19.287	31.046	8 - 7	7.527
8 - 5	16.447	26.965	37.482	8 - 5	16.447

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
4 - 8	-21.579	-9.820	1.939	4 - 8	-21.579
4 - 2	-9.355	0.395	10.146	4 - 2	-9.355
4 - 3	-7.272	2.667	12.605	4 - 3	-7.272
4 - 1	-5.323	3.901	13.126	4 - 1	-5.323
4 - 7	-2.293	9.467	21.226	4 - 7	-2.293
4 - 5	6.627	17.145	27.662	4 - 5	6.627

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
2 - 8	-19.966	-10.215	-0.465	2 - 8	-19.966
2 - 4	-10.146	-0.395	9.355	2 - 4	-10.146
2 - 3	-5.183	2.271	9.725	2 - 3	-5.183
2 - 1	-2.966	3.506	9.978	2 - 1	-2.966
2 - 7	-0.679	9.071	18.821	2 - 7	-0.679
2 - 5	8.539	16.749	24.960	2 - 5	8.539

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
3 - 8	-22.425	-12.487	-2.548	3 - 8	-22.425
3 - 4	-12.605	-2.667	7.272	3 - 4	-12.605
3 - 2	-9.725	-2.271	5.183	3 - 2	-9.725
3 - 1	-5.517	1.235	7.986	3 - 1	-5.517
3 - 7	-3.138	6.800	16.738	3 - 7	-3.138
3 - 5	6.045	14.478	22.911	3 - 5	6.045

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
1 - 8	-22.946	-13.721	-4.497	1 - 8	-22.946
1 - 4	-13.126	-3.901	5.323	1 - 4	-13.126
1 - 2	-9.978	-3.506	2.966	1 - 2	-9.978
1 - 3	-7.986	-1.235	5.517	1 - 3	-7.986
1 - 7	-3.659	5.565	14.790	1 - 7	-3.659
1 - 5	5.665	13.243	20.822	1 - 5	5.665

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit	METH Comparison	Simultaneous Lower Confidence Limit
7 - 8	-31.046	-19.287	-7.527	7 - 8	-31.046
7 - 4	-21.226	-9.467	2.293	7 - 4	-21.226
7 - 2	-18.821	-9.071	0.679	7 - 2	-18.821
7 - 3	-16.738	-6.800	3.138	7 - 3	-16.738

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cu

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 38 MSE= 15.63736
Critical Value of Studentized Range= 4.533

Comparisons significant at the 0.05 level are indicated by '***'.

		Simultaneous		Simultaneous	
		Lower	Difference	Upper	
		Confidence	Between	Confidence	
		Limit	Means	Limit	
METH	Comparison				
8	- 4	-8.337	2.013	12.363	
8	- 2	-5.692	3.272	12.235	
8	- 1	-4.315	3.660	11.635	
8	- 3	-3.066	5.681	14.428	
8	- 6	-3.403	6.947	17.297	
8	- 5	-0.653	8.605	17.862	
8	- 7	-0.103	10.247	20.597	
4	- 8	-12.363	-2.013	8.337	
4	- 2	-7.705	1.258	10.222	
4	- 1	-6.329	1.646	9.622	
4	- 3	-5.080	3.668	12.415	
4	- 6	-5.417	4.933	15.283	
4	- 5	-2.666	6.591	15.849	
4	- 7	-2.117	8.233	18.583	
2	- 8	-12.235	-3.272	5.692	
2	- 4	-10.222	-1.258	7.705	
2	- 1	-5.680	0.388	6.456	
2	- 3	-4.643	2.409	9.462	
2	- 6	-5.288	3.675	12.638	
2	- 5	-2.343	5.333	13.009	
2	- 7	-1.988	6.975	15.938	
1	- 8	-11.635	-3.660	4.315	
1	- 4	-9.622	-1.646	6.329	
1	- 2	-6.456	-0.388	5.680	
1	- 3	-3.723	2.021	7.765	
1	- 6	-4.688	3.287	11.262	
1	- 5	-1.550	4.945	11.439	
1	- 7	-1.388	6.587	14.562	
3	- 8	-14.428	-5.681	3.066	
3	- 4	-12.415	-3.668	5.080	
3	- 2	-9.462	-2.409	4.643	
3	- 1	-7.765	-2.021	3.723	
3	- 6	-7.482	1.266	10.013	
3	- 5	-4.499	2.924	10.346	
3	- 7	-4.182	4.566	13.313	

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 2 3 4 5 6 7 8

Number of observations in by group = 46

Dependent Variable: Cu

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	307.76006279	2.81	0.0184
Error	38	594.21967851		
Corrected Total	45	901.97974130		
R-Square		C.V.		Cu Mean
	0.341205	31.64904		12.49456522

Source	DF	Type I SS	F Value	Pr > F
METH	7	307.76006279	2.81	0.0184
Source	DF	Type III SS	F Value	Pr > F
METH	7	307.76006279	2.81	0.0184

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 42

Dependent Variable: Cu

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	378.90223243	8.06	0.0001
Error	35	274.39275805		
Corrected Total	41	653.29499048		

R-Square C.V. Cu Mean

0.579986 29.83067 9.38619048

Source	DF	Type I SS	F Value	Pr > F
METH	6	378.90223243	8.06	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	378.90223243	8.06	0.0001

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
	6	8	6	8	6	8
6	-17.297	-17.862	-6.947	-8.605	3.403	0.653
6	-15.283	-15.849	-4.933	-6.591	5.417	2.666
6	-12.638	-13.009	-3.675	-5.333	5.288	2.343
6	-11.262	-11.439	-3.287	-4.945	4.688	1.550
6	-10.013	-10.346	-1.266	-2.924	7.482	4.499
6	-7.599	-10.915	1.658	-1.658	10.915	7.599
6	-7.050	-7.615	3.300	1.642	13.650	10.899
5	-17.862	-17.862	-8.605	-8.605	0.653	0.653
5	-15.849	-15.849	-6.591	-6.591	2.666	2.666
5	-13.009	-13.009	-5.333	-5.333	2.343	2.343
5	-11.439	-11.439	-4.945	-4.945	1.550	1.550
5	-10.346	-10.346	-2.924	-2.924	4.499	4.499
5	-10.915	-10.915	-1.658	-1.658	7.599	7.599
5	-7.615	-7.615	1.642	1.642	10.899	10.899
7	-20.597	-20.597	-10.247	-10.247	0.103	0.103
7	-18.583	-18.583	-8.233	-8.233	2.117	2.117
7	-15.938	-15.938	-6.975	-6.975	1.988	1.988
7	-14.562	-14.562	-6.587	-6.587	1.388	1.388
7	-13.313	-13.313	-4.566	-4.566	4.182	4.182
7	-13.650	-13.650	-3.300	-3.300	7.050	7.050
7	-10.899	-10.899	-1.642	-1.642	7.615	7.615

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cu

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 7.839793
Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
5 - 1	-6.858	2.354	-2.252	2.354	-6.858	2.354
5 - 7	-3.078	9.706	3.314	9.706	-3.078	9.706
7 - 8	-19.357	-5.063	-12.210	-5.063	-19.357	-5.063
7 - 4	-17.747	-3.453	-10.600	-3.453	-17.747	-3.453
7 - 2	-14.649	-2.798	-8.724	-2.798	-14.649	-2.798
7 - 3	-11.907	0.173	-5.867	0.173	-11.907	0.173
7 - 1	-11.172	0.040	-5.566	0.040	-11.172	0.040
7 - 5	-9.706	3.078	-3.314	3.078	-9.706	3.078

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
8 - 4	-5.537	8.757	1.610	8.757	-5.537	8.757
8 - 2	-2.439	9.412	3.486	9.412	-2.439	9.412
8 - 3	0.303	12.383	6.343	12.383	0.303	12.383
8 - 1	1.038	12.250	6.644	12.250	1.038	12.250
8 - 5	2.504	15.288	8.896	15.288	2.504	15.288
8 - 7	5.063	19.357	12.210	19.357	5.063	19.357
4 - 8	-8.757	5.537	-1.610	5.537	-8.757	5.537
4 - 2	-4.049	7.802	1.876	7.802	-4.049	7.802
4 - 3	-1.307	10.773	4.733	10.773	-1.307	10.773
4 - 1	-0.572	10.640	5.034	10.640	-0.572	10.640
4 - 5	0.894	13.678	7.286	13.678	0.894	13.678
4 - 7	3.453	17.747	10.600	17.747	3.453	17.747
2 - 8	-9.412	2.439	-3.486	2.439	-9.412	2.439
2 - 4	-7.802	4.049	-1.876	4.049	-7.802	4.049
2 - 3	-1.673	7.387	2.857	7.387	-1.673	7.387
2 - 1	-0.776	7.091	3.158	7.091	-0.776	7.091
2 - 5	0.420	10.400	5.410	10.400	0.420	10.400
2 - 7	2.798	14.649	8.724	14.649	2.798	14.649
3 - 8	-12.383	-0.303	-6.343	-0.303	-12.383	-0.303
3 - 4	-10.773	1.307	-4.733	1.307	-10.773	1.307
3 - 2	-7.387	1.673	-2.857	1.673	-7.387	1.673
3 - 1	-3.802	4.404	0.301	4.404	-3.802	4.404
3 - 5	-2.572	7.678	2.553	7.678	-2.572	7.678
3 - 7	-0.173	11.907	5.867	11.907	-0.173	11.907
1 - 8	-12.250	-1.038	-6.644	-1.038	-12.250	-1.038
1 - 4	-10.640	0.572	-5.034	0.572	-10.640	0.572
1 - 2	-7.091	0.776	-3.158	0.776	-7.091	0.776
1 - 3	-4.404	3.802	-0.301	3.802	-4.404	3.802
1 - 5	-2.354	6.858	2.252	6.858	-2.354	6.858
1 - 7	-0.040	11.172	5.566	11.172	-0.040	11.172
5 - 8	-15.288	-2.504	-8.896	-2.504	-15.288	-2.504
5 - 4	-13.678	-0.894	-7.286	-0.894	-13.678	-0.894
5 - 2	-10.400	-5.410	-5.410	-5.410	-10.400	-5.410
5 - 3	-7.678	-2.553	-2.553	-2.553	-7.678	-2.553

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 41

Dependent Variable: Cu

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	93.95703506	9.18	0.0001
Error	34	57.99316982		
Corrected Total	40	151.95020488		

R-Square C.V. Cu Mean

0.618341 38.26952 3.41268293

Source	DF	Type I SS	F Value	Pr > F
METH	6	93.95703506	9.18	0.0001

Source	DF	Type III SS	F Value	Pr > F
METH	6	93.95703506	9.18	0.0001

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cu

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 34 MSE= 1.705681
Critical Value of Studentized Range= 4.428

Comparisons significant at the 0.05 level are indicated by '****'.

		Simultaneous		Difference		Simultaneous	
		Lower	Upper	Between	Confidence	Lower	Upper
		Confidence	Limit	Means	Limit	Confidence	Limit
		METH Comparison					
8	- 4	-3.123	0.217	3.556			
8	- 2	-0.027	2.795	5.617			
8	- 1	1.032	3.651	6.271			***
8	- 3	1.781	4.603	7.425			***
8	- 7	1.444	4.783	8.123			***
8	- 5	1.803	4.789	7.776			***
4	- 8	-3.556	-0.217	3.123			
4	- 2	-0.244	2.578	5.400			
4	- 1	0.815	3.434	6.054			***
4	- 3	1.565	4.387	7.209			***
4	- 7	1.227	4.567	7.906			***
4	- 5	1.586	4.573	7.559			***
2	- 8	-5.617	-2.795	0.027			
2	- 4	-5.400	-2.578	0.244			
2	- 1	-1.061	0.856	2.774			
2	- 3	-0.377	1.809	3.995			
2	- 7	-0.834	1.989	4.811			
2	- 5	-0.400	1.995	4.389			
1	- 8	-6.271	-3.651	-1.032			***
1	- 4	-6.054	-3.434	-0.815			***
1	- 2	-2.774	-0.856	1.061			
1	- 3	-0.965	0.952	2.870			
1	- 7	-1.487	1.132	3.752			
1	- 5	-1.014	1.138	3.290			
3	- 8	-7.425	-4.603	-1.781			***
3	- 4	-7.209	-4.387	-1.565			***
3	- 2	-3.995	-1.809	0.377			
3	- 1	-2.870	-0.952	0.965			
3	- 7	-2.642	0.180	3.002			
3	- 5	-2.209	0.186	2.581			
7	- 8	-8.123	-4.783	-1.444			***
7	- 4	-7.906	-4.567	-1.227			***
7	- 2	-4.811	-1.989	0.834			
7	- 1	-3.752	-1.132	1.487			

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
7 - 3	-3.002	-0.180	2.642	
7 - 5	-2.981	0.006	2.993	
5 - 8	-7.776	-4.789	-1.803	***
5 - 4	-7.559	-4.573	-1.586	***
5 - 2	-4.389	-1.995	0.400	
5 - 1	-3.290	-1.138	1.014	
5 - 3	-2.581	-0.186	2.209	
5 - 7	-2.993	-0.006	2.981	

units: mg/kg
optional for all samples
reported by 11 laboratories

TABLE I : Pb - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N/R
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	R
11			
12			
13			
14	A1 A2 A3 B1 B2	5	N
15	A1 A2 B1 B2	3	N
16	A1 A2 B1 B2	3	N/R
17			
18	A1 A2 B1 B2	3	N/R
19			
20			
21			
22			

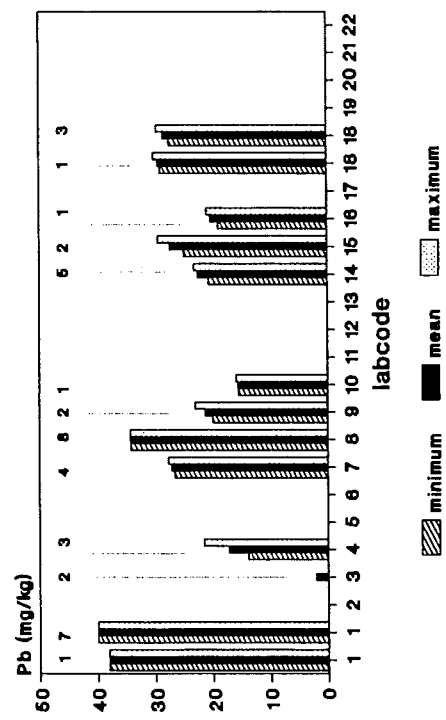
(*) R = aqua regia

TABLE II : Pb - Summary method descriptions

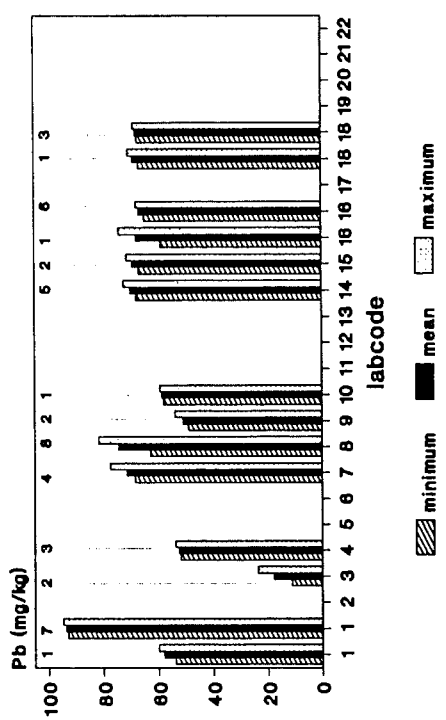
method	description
1	aqua regia
2	total ICP-AES, HNO3 + HClO4
3	total analysis, 2M HNO3
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO3 under pressure
7	2N HCl
8	minéralisation
* 9	unspecified

* not applicable

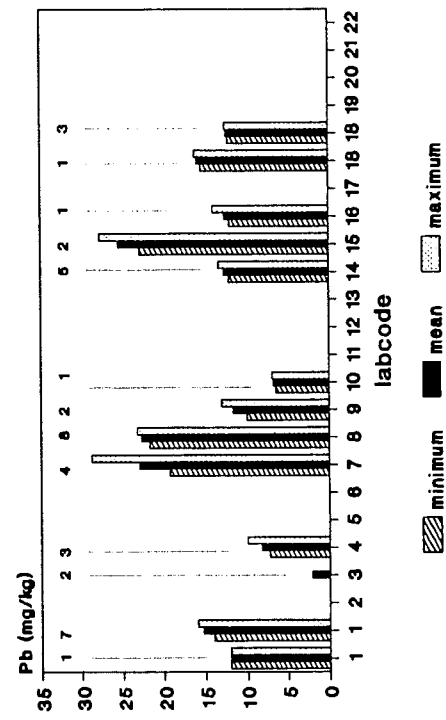
Pb sample A1
national/reference methods



Pb sample A2
national/reference methods



Pb sample B1
national/reference methods



Pb sample B2
national/reference methods

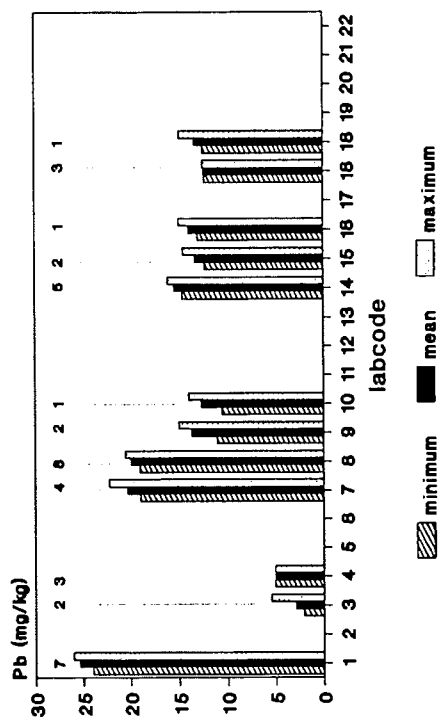


TABLE III : Pb - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	38.00	38.00	38.00	0.00	0.00
1	7	3	40.00	40.00	40.00	0.00	0.00
2							
3	2	5		2.07			
4	3	4	13.90	17.28	21.50	3.28	18.98
5							
6							
7	4	3	26.70	27.20	27.80	0.56	2.05
8	8	3	34.16	34.26	34.33	0.09	0.27
9	2	3	20.00	21.33	23.00	1.53	7.16
10	1	3	15.50	15.67	16.00	0.29	1.84
11							
12							
13							
14	5	5	20.70	22.48	23.20	1.03	4.58
15	2	4	25.02	27.46	29.51	1.87	6.79
16	1	3	19.00	20.33	21.00	1.16	5.68
17							
18	1	3	29.06	29.53	30.28	0.65	2.22
18	3	3	27.53	28.58	29.67	1.07	3.75
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	54.00	58.00	60.00	3.46	5.97
1	7	3	93.00	93.67	95.00	1.16	1.23
2							
3	2	5	10.86	17.73	23.74	5.04	28.43
4	3	4	51.90	52.65	53.60	0.73	1.39
5							
6							
7	4	3	68.30	71.37	77.20	5.05	7.08
8	8	3	62.92	74.41	81.74	10.08	13.54
9	2	3	49.00	51.00	54.00	2.65	5.19
10	1	3	58.00	58.67	59.50	0.76	1.30
11							
12							
13							
14	5	5	68.00	70.04	72.40	1.91	2.73
14	A3	5	69.00	70.34	71.90	1.04	1.48
15	2	3	66.90	69.33	71.40	2.27	3.28
16	1	3	59.00	68.00	74.00	7.94	11.67
16	6	3	65.00	67.00	68.00	1.73	2.59
17							
18	1	3	66.96	69.06	70.55	1.87	2.71
18	3	3	67.39	68.08	68.81	0.71	1.04
19							
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	12.00	12.00	12.00	0.00	0.00
1	7	3	14.00	15.33	16.00	1.16	7.53
2							
3	2	5		2.07			
4	3	4	7.31	8.25	9.94	1.18	14.23
5							
6							
7	4	3	19.30	22.83	28.80	5.20	22.76
8	8	3	21.67	22.61	23.20	0.82	3.63
9	2	3	10.00	11.67	13.00	1.53	13.09
10	1	3	6.50	6.83	7.00	0.29	4.23
11							
12							
13							
14	5	5	12.10	12.70	13.30	0.50	3.90
15	2	4	22.80	25.57	27.80	2.14	8.37
16	1	3	12.00	12.67	14.00	1.16	9.12
17							
18	1	3	15.43	15.94	16.24	0.44	2.77
18	3	3	12.18	12.38	12.59	0.21	1.66
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	38.00	38.00	38.00	0.00	0.00
1	7	3	24.00	25.33	26.00	1.16	4.56
2							
3	2	5	2.07	2.86	5.51	1.40	48.95
4	3	4	5.08	5.08	5.08	0.00	0.00
5							
6							
7	4	3	18.90	20.33	22.30	1.76	8.66
8	8	3	18.97	19.94	20.50	0.84	4.22
9	2	3	11.00	13.67	15.00	2.31	16.90
10	1	3	10.50	12.67	14.00	1.89	14.94
11							
12							
13							
14	5	5	14.60	15.46	16.10	0.63	4.08
15	2	4	12.30	13.33	14.55	1.01	7.55
16	1	3	13.00	14.00	15.00	1.00	7.14
17							
18	3	3	12.25	12.32	12.45	0.12	0.94
18	1	3	12.45	13.29	14.98	1.46	10.99
19							
20							
21							
22							

TABLE V : Pb Method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 41

Dependent Variable: Pb

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	1091.72821998	3.68	0.0063
Error	34	1681.29041905		
Corrected Total	40	2773.01863902		

R-Square	C.V.	Pb Mean
0.393697	27.24673	25.80878049

Source	DF	Type I SS	F Value	Pr > F
METH	6	1091.72821998	3.68	0.0063
Source	DF	Type III SS	F Value	Pr > F
METH	6	1091.72821998	3.68	0.0063

TABLE IV : Pb - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<w
A1	1	12	25.88	8.99	0.87	0.07
A1	2	8	21.99	8.73	0.78	0.02
A1	3	7	22.12	6.50	0.89	0.29
A1	4	3	27.20	0.56	0.98	0.70
A1	5	5	22.48	1.03	0.77	0.04
A1	7	3	40.00	0.00		
A1	8	3	34.26	0.09	0.88	0.32
A2	1	12	63.43	6.55	0.89	0.12
A2	2	7	54.10	18.58	0.86	0.14
A2	3	7	59.26	8.28	0.73	0.01
A2	4	3	71.37	5.05	0.78	0.06
A2	5	5	70.04	1.91	0.93	0.57
A2	6	3	67.00	1.73	0.75	0.00
A2	7	3	93.67	1.16	0.75	0.00
A2	8	3	74.41	10.08	0.87	0.30
A3	5	5	70.34	1.04	0.95	0.71
B1	1	12	11.86	3.45	0.87	0.07
B1	2	8	17.42	9.40	0.91	0.32
B1	3	7	10.02	2.36	0.83	0.09
B1	4	3	22.83	5.20	0.84	0.20
B1	5	5	12.70	0.50	0.95	0.76
B1	7	3	15.33	1.16	0.75	0.00
B1	8	3	22.61	0.82	0.87	0.29
B2	1	9	13.32	1.42	0.93	0.49
B2	2	8	12.15	4.01	0.73	0.01
B2	3	7	8.18	3.87	0.67	0.00
B2	4	3	20.33	1.76	0.93	0.49
B2	5	5	15.46	0.63	0.89	0.35
B2	7	3	25.33	1.16	0.75	0.00
B2	8	3	19.94	0.84	0.83	0.18

Remark:

Labcode 3 reports the mean values for 5 replicates.
To calculate the total number of replicates for the method-grouped laboratories,
SAS counts the number of reported results.
So, the "repl" values in this table for method 2 are different from the real number
of replicates.

SAMPLE=A1

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
3 - 5	-13.252		-0.359		12.535	
3 - 2	-11.265		0.131		11.528	
2 - 7	-32.918		-18.010		-3.102	***
2 - 8	-27.181		-12.273		2.634	
2 - 4	-20.118		-5.210		9.698	
2 - 1	-13.944		-3.893		6.157	
2 - 5	-13.043		-0.490		12.063	
2 - 3	-11.528		-0.131		11.265	

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Pb

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 34 MSE= 49.44972
Critical Value of Studentized Range= 4.428

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit	
7 - 8	-12.243		5.737		23.716	
7 - 4	-5.179		12.800		30.779	
7 - 1	-0.097		14.117		28.331	***
7 - 5	1.439		17.520		33.601	***
7 - 3	2.683		17.879		33.074	***
7 - 2	3.102		18.010		32.918	***
8 - 7	-23.716		-5.737		12.243	
8 - 4	-10.916		7.063		25.043	
8 - 1	-5.834		8.380		22.594	
8 - 5	-4.298		11.783		27.865	
8 - 3	-3.053		12.142		27.337	
8 - 2	-2.634		12.273		27.181	
4 - 7	-30.779		-12.800		5.179	
4 - 8	-25.043		-7.063		10.916	
4 - 1	-12.897		1.317		15.531	
4 - 5	-11.361		4.720		20.801	
4 - 3	-10.117		5.079		20.274	
4 - 2	-9.698		5.210		20.118	
1 - 7	-28.331		-14.117		0.097	
1 - 8	-22.594		-8.380		5.834	
1 - 4	-15.531		-1.317		12.897	
1 - 5	-8.318		3.403		15.124	
1 - 3	-6.711		3.762		14.235	
1 - 2	-6.157		3.893		13.944	
5 - 7	-33.601		-17.520		-1.439	***
5 - 8	-27.865		-11.783		4.298	
5 - 4	-20.801		-4.720		11.361	
5 - 1	-15.124		-3.403		8.318	
5 - 3	-12.535		0.359		13.252	
5 - 2	-12.063		0.490		13.043	
3 - 7	-33.074		-17.879		-2.683	***
3 - 8	-27.337		-12.142		3.053	
3 - 4	-20.274		-5.079		10.117	
3 - 1	-14.235		-3.762		6.711	

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Pb

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 92.37324
Critical Value of Studentized Range= 4.555

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7	- 8	-6.023	19.253	19.253	44.529	44.529
7	- 4	-2.976	22.300	22.300	47.576	47.576
7	- 5	1.019	23.627	23.627	46.234	***
7	- 6	1.391	26.667	26.667	51.943	***
7	- 1	10.252	30.234	30.234	50.216	***
7	- 3	13.040	34.402	34.402	55.764	***
7	- 2	18.200	39.562	39.562	60.924	***
8	- 7	-44.529	-19.253	-19.253	6.023	
8	- 4	-22.229	3.047	3.047	28.323	
8	- 5	-18.234	4.373	4.373	26.981	
8	- 6	-17.863	7.413	7.413	32.689	
8	- 1	-9.001	10.981	10.981	30.963	
8	- 3	-6.213	15.149	15.149	36.511	
8	- 2	-1.053	20.309	20.309	41.671	
4	- 7	-47.576	-22.300	-22.300	2.976	
4	- 8	-28.323	-3.047	-3.047	22.229	
4	- 5	-21.281	1.327	1.327	23.934	
4	- 6	-20.909	4.367	4.367	29.643	
4	- 1	-12.048	7.934	7.934	27.916	
4	- 3	-9.260	12.102	12.102	33.464	
4	- 2	-4.100	17.262	17.262	38.624	
5	- 7	-46.234	-23.627	-23.627	-1.019	***
5	- 8	-26.981	-4.373	-4.373	18.234	
5	- 4	-23.934	-1.327	-1.327	21.281	
5	- 6	-19.567	3.040	3.040	25.647	
5	- 1	-9.870	6.608	6.608	23.085	
5	- 3	-7.351	10.776	10.776	28.902	
5	- 2	-2.191	15.936	15.936	34.062	
6	- 7	-51.943	-26.667	-26.667	-1.391	***
6	- 8	-32.689	-7.413	-7.413	17.863	
6	- 4	-29.643	-4.367	-4.367	20.909	
6	- 5	-25.647	-3.040	-3.040	19.567	
6	- 1	-16.415	3.567	3.567	23.550	
6	- 3	-13.626	7.736	7.736	29.098	
6	- 2	-8.466	12.896	12.896	34.258	

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class Levels Values

METH 8 1 2 3 4 5 6 7 8

Number of observations in by group = 43

Dependent Variable: Pb

Source	DF	Sum of Squares	F Value	Pr > F
Model	7	4062.58841819	6.28	0.0001
Error	35	3233.06356786		
Corrected Total	42	7295.65198605		

R-Square C.V. Pb Mean

0.556851 14.63286 65.68162791

Source	DF	Type I SS	F Value	Pr > F
METH	7	4062.58841819	6.28	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	7	4062.58841819	6.28	0.0001

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 41

Dependent Variable: Pb

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	715.62796670	4.81	0.0012
Error	34	842.24038452		
Corrected Total	40	1557.86835122		
R-Square C.V. Pb Mean				
		34.14523		14.57634146
Type I SS F Value Pr > F				
Source	DF	Type I SS	F Value	Pr > F
METH	6	715.62796670	4.81	0.0012
Type III SS F Value Pr > F				
Source	DF	Type III SS	F Value	Pr > F
METH	6	715.62796670	4.81	0.0012

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
1 - 7	-50.216	-30.234	-10.252	***
1 - 8	-30.963	-10.981	9.001	
1 - 4	-27.916	-7.934	12.048	
1 - 5	-23.085	-6.608	9.870	
1 - 6	-23.550	-3.567	16.415	
1 - 3	-10.555	4.168	18.891	
1 - 2	-5.395	9.328	24.051	
3 - 7	-55.764	-34.402	-13.040	***
3 - 8	-36.511	-15.149	6.213	
3 - 4	-33.464	-12.102	9.260	
3 - 5	-28.902	-10.776	7.351	
3 - 6	-29.098	-7.736	13.626	
3 - 1	-18.891	-4.168	10.555	
3 - 2	-11.387	5.160	21.707	
2 - 7	-60.924	-39.562	-18.200	***
2 - 8	-41.671	-20.309	1.053	
2 - 4	-38.624	-17.262	4.100	
2 - 5	-34.062	-15.936	2.191	
2 - 6	-34.258	-12.896	8.466	
2 - 1	-24.051	-9.328	5.395	
2 - 3	-21.707	-5.160	11.387	

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Pb

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 34 MSE= 24.77178
Critical Value of Studentized Range= 4.428

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Lower Confidence Limit			Simultaneous Upper Confidence Limit		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 5	-9.137	-0.841	7.455	-9.137	-0.841	7.455
1 - 3	-5.576	1.836	9.249	-5.576	1.836	9.249
3 - 4	-23.565	-12.810	-2.056	-23.565	-12.810	-2.056
3 - 8	-23.339	-12.584	-1.829	-23.339	-12.584	-1.829
3 - 2	-15.461	-7.395	0.672	-15.461	-7.395	0.672
3 - 7	-16.065	-5.310	5.444	-16.065	-5.310	5.444
3 - 5	-11.803	-2.677	6.449	-11.803	-2.677	6.449
3 - 1	-9.249	-1.836	5.576	-9.249	-1.836	5.576

METH Comparison	Simultaneous Lower Confidence Limit			Simultaneous Upper Confidence Limit		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 8	-12.499	0.227	12.952	-12.499	0.227	12.952
4 - 2	-5.136	5.416	15.967	-5.136	5.416	15.967
4 - 7	-5.225	7.500	20.225	-5.225	7.500	20.225
4 - 5	-1.249	10.133	21.515	-1.249	10.133	21.515
4 - 1	0.914	10.974	21.034	0.914	10.974	21.034
4 - 3	2.056	12.810	23.565	2.056	12.810	23.565
8 - 4	-12.952	-0.227	12.499	-12.952	-0.227	12.499
8 - 2	-5.362	5.189	15.741	-5.362	5.189	15.741
8 - 7	-5.452	7.273	19.999	-5.452	7.273	19.999
8 - 5	-1.475	9.907	21.289	-1.475	9.907	21.289
8 - 1	0.687	10.748	20.808	0.687	10.748	20.808
8 - 3	1.829	12.584	23.339	1.829	12.584	23.339
2 - 4	-15.967	-5.416	5.136	-15.967	-5.416	5.136
2 - 8	-15.741	-5.189	5.362	-15.741	-5.189	5.362
2 - 7	-8.467	2.084	12.636	-8.467	2.084	12.636
2 - 5	-4.168	4.718	13.603	-4.168	4.718	13.603
2 - 1	-1.555	5.558	12.672	-1.555	5.558	12.672
2 - 3	-0.672	7.395	15.461	-0.672	7.395	15.461
7 - 4	-20.225	-7.500	5.225	-20.225	-7.500	5.225
7 - 8	-19.999	-7.273	5.452	-19.999	-7.273	5.452
7 - 2	-12.636	-2.084	8.467	-12.636	-2.084	8.467
7 - 5	-8.749	2.633	14.015	-8.749	2.633	14.015
7 - 1	-6.586	3.474	13.534	-6.586	3.474	13.534
7 - 3	-5.444	5.310	16.065	-5.444	5.310	16.065
5 - 4	-21.515	-10.133	1.249	-21.515	-10.133	1.249
5 - 8	-21.289	-9.907	1.475	-21.289	-9.907	1.475
5 - 2	-13.603	-4.718	4.168	-13.603	-4.718	4.168
5 - 7	-14.015	-2.633	8.749	-14.015	-2.633	8.749
5 - 1	-7.455	0.841	9.137	-7.455	0.841	9.137
5 - 3	-6.449	2.677	11.803	-6.449	2.677	11.803
1 - 4	-21.034	-10.974	-0.914	-21.034	-10.974	-0.914
1 - 8	-20.808	-10.748	-0.687	-20.808	-10.748	-0.687
1 - 2	-12.672	-5.558	1.555	-12.672	-5.558	1.555
1 - 7	-13.534	-3.474	6.586	-13.534	-3.474	6.586

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Pb

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 31 MSE= 7.427911
Critical Value of Studentized Range= 4.454

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7	- 4	-2.009	5.000	5.000	12.009	12.009
7	- 8	-1.612	5.397	5.397	12.406	12.406
7	- 5	3.604	9.873	9.873	16.142	16.142
7	- 1	6.290	12.013	12.013	17.736	17.736
7	- 2	7.374	13.186	13.186	18.997	18.997
7	- 3	11.228	17.152	17.152	23.076	23.076
4	- 7	-12.009	-5.000	-5.000	2.009	2.009
4	- 8	-6.612	0.397	0.397	7.406	7.406
4	- 5	-1.396	4.873	4.873	11.142	11.142
4	- 1	1.290	7.013	7.013	12.736	12.736
4	- 2	2.374	8.186	8.186	13.997	13.997
4	- 3	6.228	12.152	12.152	18.076	18.076
8	- 7	-12.406	-5.397	-5.397	1.612	1.612
8	- 4	-7.406	-0.397	-0.397	6.612	6.612
8	- 5	-1.792	4.477	4.477	10.746	10.746
8	- 1	0.894	6.617	6.617	12.340	12.340
8	- 2	1.978	7.789	7.789	13.601	13.601
8	- 3	5.832	11.755	11.755	17.679	17.679
5	- 7	-16.142	-9.873	-9.873	-3.604	-3.604
5	- 4	-11.142	-4.873	-4.873	1.396	1.396
5	- 8	-10.746	-4.477	-4.477	1.792	1.792
5	- 1	-2.648	2.140	2.140	6.928	6.928
5	- 2	-1.581	3.313	3.313	8.206	8.206
5	- 3	2.252	7.279	7.279	12.305	12.305
1	- 7	-17.736	-12.013	-12.013	-6.290	-6.290
1	- 4	-12.736	-7.013	-7.013	-1.290	-1.290
1	- 8	-12.340	-6.617	-6.617	-0.894	-0.894
1	- 5	-6.928	-2.140	-2.140	2.648	2.648
1	- 2	-2.999	1.173	1.173	5.344	5.344
1	- 3	0.813	5.139	5.139	9.465	9.465
2	- 7	-18.997	-13.186	-13.186	-7.374	-7.374
2	- 4	-13.997	-8.186	-8.186	-2.374	-2.374
2	- 8	-13.601	-7.789	-7.789	-1.978	-1.978
2	- 5	-8.206	-3.313	-3.313	1.581	1.581

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 41

NOTE: Due to missing values, only 38 observations can be used in this analysis.

Dependent Variable: Pb				
Source	DF	Sum of Squares	F Value	Pr > F
Model	6	883.55030113	19.83	0.0001
Error	31	230.26523571		
Corrected Total	37	1113.81553684		
R-Square		C.V.	Pb Mean	
	0.793264	18.88373	14.43263158	
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	6	883.55030113	19.83	0.0001
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	6	883.55030113	19.83	0.0001

----- SAMPLE=B2 -----

General Linear Models Procedure

METH Comparison	Simultaneous		Simultaneous	
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	
2 - 1	-5.344	-1.173	2.999	
2 - 3	-0.477	3.966	8.409	
3 - 7	-23.076	-17.152	-11.228	***
3 - 4	-18.076	-12.152	-6.228	***
3 - 8	-17.679	-11.755	-5.832	***
3 - 5	-12.305	-7.279	-2.252	***
3 - 1	-9.465	-5.139	-0.813	***
3 - 2	-8.409	-3.966	0.477	

32. Mn

units: mg/kg
optional for all samples
reported by 13 laboratories

TABLE I : Mn - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1	A1 A2 B1 B2	3	N/R
2			
3	A1 A2 B1 B2	5	N
4	A1 A2 B1 B2	4	N
5			
6			
7	A1 A2 B1 B2	3	N
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	N
11			
12			
13			
14	A1 A2 A3 B1 B2	4	N
15	A1 A2 B1 B2	A1 B1 B2:4 A2:2	N
16	A1 A2 B1 B2	3	N/R
17			
18	A1 A2 B1 B2	3	N/R
19	A1 A2 B1 B2	3	R
20			
21			
22	A2	3	R

(*) R = aqua regia

TABLE II : Mn - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HNO3 + HClO4
3	total analysis, 2M HNO3
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO3 under pressure
7	2N HCl
8	minéralisation

TABLE III : Mn - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	439.00	439.00	439.00	0.00	0.00
1	7	3	327.00	327.00	327.00	0.00	0.00
2							
3	2	5	547.18	552.13	555.42	3.30	0.60
4	3	4	474.00	511.50	567.00	45.73	8.94
5							
6							
7	4	3	716.00	716.33	717.00	0.58	0.08
8	8	3	482.50	487.50	490.00	4.33	0.89
9	2	3	607.00	610.00	615.00	4.36	0.72
10	1	3	527.00	533.33	538.00	5.69	1.07
11							
12							
13							
14	5	5	392.00	456.00	498.00	38.90	8.53
15	2	4	437.50	472.20	499.00	25.61	5.42
16	1	3	508.00	509.33	511.00	1.53	0.30
17							
18	1	3	636.02	641.10	647.19	5.65	0.88
18	3	3	588.26	590.29	591.31	1.76	0.30
19	1	3		712.00			
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	500.00	503.67	508.00	4.04	0.80
1	7	3	544.00	552.00	557.00	7.00	1.27
2							
3	2	5	799.90	819.13	828.47	10.99	1.34
4	3	4	798.70	808.10	827.10	13.13	1.63
5							
6							
7	4	3	810.00	819.33	837.00	15.31	1.87
8	8	3	634.00	642.50	652.50	9.34	1.45
9	2	3	774.00	797.33	820.00	23.01	2.89
10	1	3	700.00	703.33	710.00	5.77	0.82
11							
12							
13							
14	5	5	751.00	765.80	774.00	9.26	1.21
14	A3	5	748.00	761.00	768.00	8.00	1.05
15	2	2	704.30	716.35	728.40	17.04	2.38
16	1	3	674.00	688.33	703.00	14.50	2.11
16	6	3	762.00	770.67	776.00	7.57	0.98
17							
18	1	3	794.60	807.64	818.51	12.10	1.50
18	3	3	767.42	769.60	772.86	2.88	0.37
19	1	3		801.00			
20							
21							
22	1	3	772.00	776.33	779.00	3.79	0.49

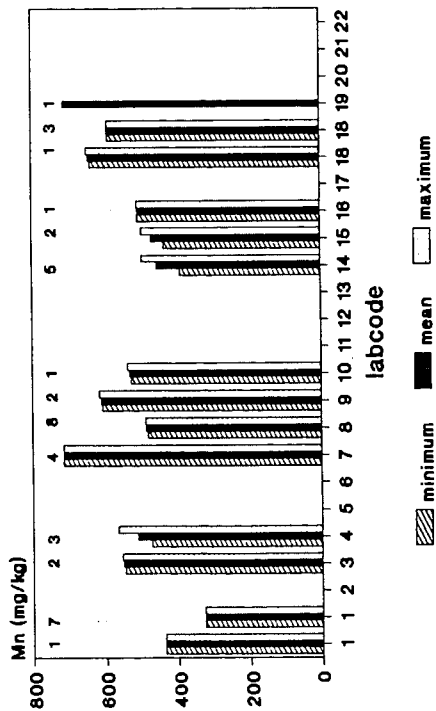
sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	245.00	247.67	249.00	2.31	0.93
1	7	3	65.00	65.00	65.00	0.00	0.00
2							
3	2	5	272.49	288.97	341.16	26.92	9.32
4	3	4	244.00	256.50	266.00	9.18	3.58
5							
6							
7	4	3	357.00	370.00	379.00	11.53	3.12
8	8	3	265.00	270.67	282.00	9.82	3.63
9	2	3	268.00	275.33	282.00	7.02	2.55
10	1	3	249.00	260.00	276.00	14.18	5.45
11							
12							
13							
14	5	5	239.00	249.80	262.00	8.82	3.53
15	2	4	215.10	228.83	254.00	17.84	7.80
16	1	3	261.00	266.00	269.00	4.36	1.64
17							
18	1	3	275.07	282.85	289.28	7.20	2.55
18	3	3	259.84	260.18	260.86	0.59	0.23
19	1	3		283.00			
20							
21							
22							

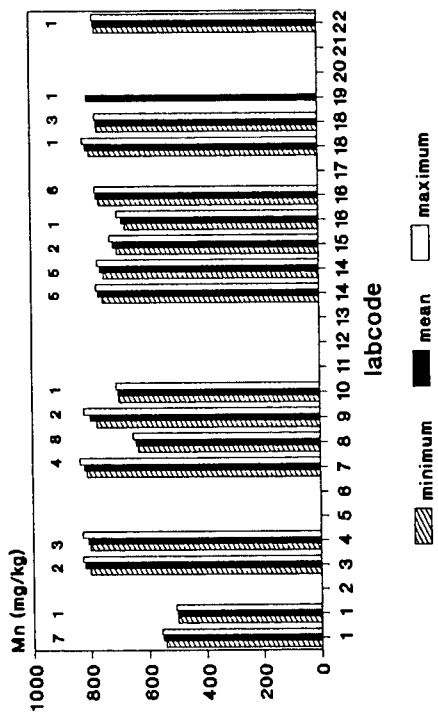
sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1	1	3	18.00	18.00	18.00	0.00	0.00
1	7	3	6.00	6.00	6.00	0.00	0.00
2							
3	2	5	9.89	10.99	13.19	1.10	10.01
4	3	4	7.11	7.45	7.62	0.23	3.14
5							
6							
7	4	3	36.70	48.97	59.20	11.39	23.26
8	8	3	30.00	31.36	33.05	1.55	4.95
9	2	3	11.00	11.67	13.00	1.16	9.90
10	1	3	9.50	9.67	10.00	0.29	2.99
11							
12							
13							
14	5	5	8.70	9.36	9.59	0.37	3.98
15	2	4	6.57	7.02	7.34	0.33	4.65
16	1	3	11.00	11.33	12.00	0.58	5.09
17							
18	3	3	6.27	6.41	6.58	0.16	2.45
18	1	3	9.31	9.55	9.72	0.21	2.22
19	1	3		12.00			
20							
21							
22							

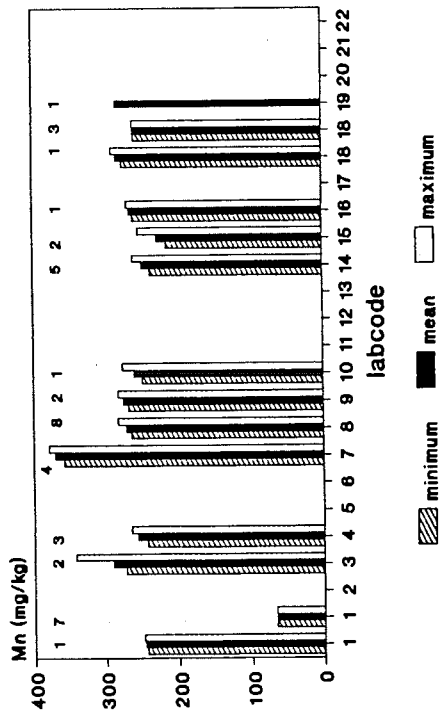
Mn sample A1
national/reference methods



Mn sample A2
national/reference methods



Mn sample B1
national/reference methods



Mn sample B2
national/reference methods

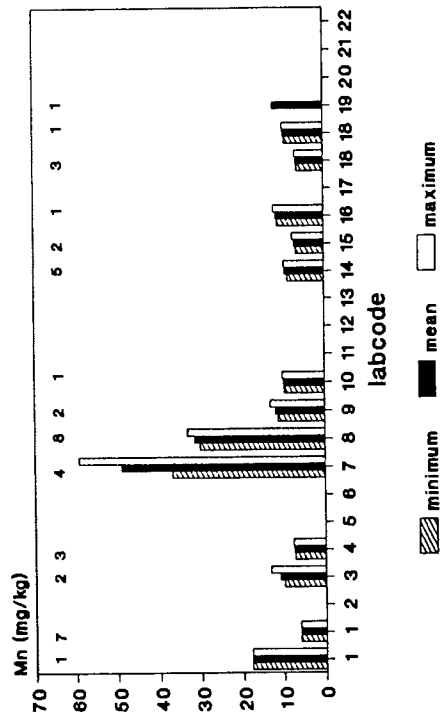


TABLE V :

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 42

Dependent Variable: Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	263976.874020	10.16	0.0001
Error	35	151610.351771		

Corrected Total

R-Square

C.V.

Mn Mean

0.635190

12.54170

524.77619048

Source

DF

Type I SS

F Value

Pr > F

METH

6

263976.874020

10.16

0.0001

Source

DF

Type III SS

F Value

Pr > F

METH

6

263976.874020

10.16

0.0001

TABLE IV : Mn - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	13	544.64	88.34	0.90	0.14
A1	2	8	533.87	70.65	0.88	0.20
A1	3	7	545.27	53.11	0.80	0.04
A1	4	3	716.33	0.58	0.75	0.00
A1	5	5	456.00	38.90	0.87	0.25
A1	7	3	327.00	0.00		
A1	8	3	487.50	4.33	0.75	0.00
A2	1	16	702.43	109.32	0.83	0.01
A2	2	6	773.97	48.30	0.89	0.32
A2	3	7	791.60	22.64	0.90	0.32
A2	4	3	819.33	15.31	0.78	0.06
A2	5	5	765.80	9.26	0.89	0.35
A2	6	3	770.67	7.57	0.86	0.25
A2	7	3	552.00	7.00	0.86	0.27
A2	8	3	642.50	9.34	0.98	0.73
A3	5	5	761.00	8.00	0.88	0.29
B1	1	13	265.58	15.30	0.93	0.29
B1	2	8	253.78	29.71	0.89	0.25
B1	3	7	258.08	6.79	0.82	0.06
B1	4	3	370.00	11.53	0.91	0.42
B1	5	5	249.80	8.82	0.98	0.92
B1	7	3	65.00	0.00		
B1	8	3	270.67	9.82	0.75	
B2	1	13	12.13	3.47	0.73	0.00
B2	2	8	9.26	2.49	0.84	0.07
B2	3	7	7.01	0.59	0.85	0.13
B2	4	3	48.97	11.39	0.98	0.70
B2	5	5	9.36	0.37	0.71	0.01
B2	7	3	6.00	0.00		
B2	8	3	31.36	1.55	0.97	0.64

Remark:

Labcode 3 and 19 reported mean values and the number of replicates,

respectively 5 and 3.

To calculate the total number of replicates for the method-grouped laboratories,

SAS counts the number of reported results.

So, the "repl" values in this table for the methods 1 and 2 are different from the real number of replicates.

SAMPLE=A1

SAMPLE=A1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 4331.724
Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
5 - 8	-181.75	-31.50	118.75	-181.75	-31.50	118.75
5 - 7	-21.25	129.00	279.25	-21.25	129.00	279.25
7 - 4	-557.32	-389.33	-221.35	-557.32	-389.33	-221.35
7 - 3	-360.24	-218.27	-76.29	-360.24	-218.27	-76.29
7 - 1	-349.42	-217.64	-85.86	-349.42	-217.64	-85.86
7 - 2	-346.15	-206.87	-67.58	-346.15	-206.87	-67.58
7 - 8	-328.49	-160.50	7.49	-328.49	-160.50	7.49
7 - 5	-279.25	-129.00	21.25	-279.25	-129.00	21.25

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
4 - 3	29.09	171.06	313.04	313.04	171.06	313.04
4 - 1	39.92	171.70	303.48	303.48	171.70	303.48
4 - 2	43.18	182.47	321.76	321.76	182.47	321.76
4 - 8	60.85	228.83	396.82	396.82	228.83	396.82
4 - 5	110.08	260.33	410.59	410.59	260.33	410.59
4 - 7	221.35	389.33	557.32	557.32	389.33	557.32
3 - 4	-313.04	-171.06	-29.09	-29.09	-171.06	-29.09
3 - 1	-95.82	0.63	97.08	97.08	0.63	97.08
3 - 2	-95.08	11.40	117.88	117.88	11.40	117.88
3 - 8	-84.21	57.77	199.74	199.74	57.77	199.74
3 - 5	-31.20	89.27	209.74	209.74	89.27	209.74
3 - 7	76.29	218.27	360.24	360.24	218.27	360.24
1 - 4	-303.48	-171.70	-39.92	-39.92	-171.70	-39.92
1 - 3	-97.08	-0.63	95.82	95.82	-0.63	95.82
1 - 2	-81.68	10.77	103.22	103.22	10.77	103.22
1 - 8	-74.64	57.14	188.92	188.92	57.14	188.92
1 - 5	-19.63	88.64	196.91	196.91	88.64	196.91
1 - 7	85.86	217.64	349.42	349.42	217.64	349.42
2 - 4	-321.76	-182.47	-43.18	-43.18	-182.47	-43.18
2 - 3	-117.88	-11.40	95.08	95.08	-11.40	95.08
2 - 1	-103.22	-10.77	81.68	81.68	-10.77	81.68
2 - 8	-92.92	46.37	185.65	185.65	46.37	185.65
2 - 5	-39.42	77.87	195.16	195.16	77.87	195.16
2 - 7	67.58	206.87	346.15	346.15	206.87	346.15
8 - 4	-396.82	-228.83	-60.85	-60.85	-228.83	-60.85
8 - 3	-199.74	-57.77	84.21	84.21	-57.77	84.21
8 - 1	-188.92	-57.14	74.64	74.64	-57.14	74.64
8 - 2	-185.65	-46.37	92.92	92.92	-46.37	92.92
8 - 5	-118.75	31.50	181.75	181.75	31.50	181.75
8 - 7	-7.49	160.50	328.49	328.49	160.50	328.49
5 - 4	-410.59	-260.33	-110.08	-110.08	-260.33	-110.08
5 - 3	-209.74	-89.27	31.20	31.20	-89.27	31.20
5 - 1	-196.91	-88.64	19.63	19.63	-88.64	19.63
5 - 2	-195.16	-77.87	39.42	39.42	-77.87	39.42

SAMPLE=A2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 38 MSE= 5137.089

Critical Value of Studentized Range= 4.533

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous Lower Limit	Simultaneous Upper Limit	Difference Between Means	Simultaneous Lower Limit	Simultaneous Upper Limit
4	- 3	-130.81	186.28	27.73	-130.81	186.28
4	- 2	-117.10	207.82	45.36	-117.10	207.82
4	- 6	-138.92	236.26	48.67	-138.92	236.26
4	- 5	-114.25	221.32	53.53	-114.25	221.32
4	- 1	-27.65	261.45	116.90	-27.65	261.45
4	- 8	-10.76	364.42	176.83	-10.76	364.42
4	- 7	79.74	454.92	267.33	79.74	454.92
3	- 4	-186.28	130.81	-27.73	-186.28	130.81
3	- 2	-110.19	145.45	17.63	-110.19	145.45
3	- 6	-137.61	179.47	20.93	-137.61	179.47
3	- 5	-108.73	160.33	25.80	-108.73	160.33
3	- 1	-14.95	193.28	89.17	-14.95	193.28
3	- 8	-9.44	307.64	149.10	-9.44	307.64
3	- 7	81.06	398.14	239.60	81.06	398.14
2	- 4	-207.82	117.10	-45.36	-207.82	117.10
2	- 3	-145.45	110.19	-17.63	-145.45	110.19
2	- 6	-159.15	165.76	3.31	-159.15	165.76
2	- 5	-130.95	147.29	8.17	-130.95	147.29
2	- 1	-38.45	181.52	71.54	-38.45	181.52
2	- 8	-30.99	293.93	131.47	-30.99	293.93
2	- 7	59.51	384.43	221.97	59.51	384.43
6	- 4	-236.26	138.92	-48.67	-236.26	138.92
6	- 3	-179.47	137.61	-20.93	-179.47	137.61
6	- 2	-165.76	159.15	-3.31	-165.76	159.15
6	- 5	-162.92	172.65	4.87	-162.92	172.65
6	- 1	-76.31	212.78	68.23	-76.31	212.78
6	- 8	-59.42	315.76	128.17	-59.42	315.76
6	- 7	31.08	406.26	218.67	31.08	406.26
5	- 4	-221.32	114.25	-53.53	-221.32	114.25
5	- 3	-160.33	108.73	-25.80	-160.33	108.73
5	- 2	-147.29	130.95	-8.17	-147.29	130.95
5	- 6	-172.65	162.92	-4.87	-172.65	162.92
5	- 1	-54.34	181.08	63.37	-54.34	181.08
5	- 8	-44.49	291.09	123.30	-44.49	291.09
5	- 7	46.01	381.59	213.80	46.01	381.59

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	8	1 2 3 4 5 6 7 8

Number of observations in by group = 46

Dependent Variable: Mn				
Source	DF	Sum of Squares	F Value	Pr > F
Model	7	203637.625145	5.66	0.0002
Error	38	195209.366802		
Corrected Total	45	398846.991948		
R-Square		C.V.	Mn Mean	
	0.510566	9.810561	730.57478261	
Source	DF	Type I SS	F Value	Pr > F
METH	7	203637.625145	5.66	0.0002
Source	DF	Type III SS	F Value	Pr > F
METH	7	203637.625145	5.66	0.0002

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 7 8

Number of observations in by group = 42

Dependent Variable: Mn

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	150308.949873	87.40	0.0001
Error	35	10032.191089		
Corrected Total	41	160341.140962		

R-Square C.V. Mn Mean

0.937432 6.673367 253.69904762

Source	DF	Type I SS	F Value	Pr > F
METH	6	150308.949873	87.40	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	6	150308.949873	87.40	0.0001

SAMPLE=A2

General Linear Models Procedure

METH Comparison	Simultaneous Lower Confidence Limit	Difference Between Means	Simultaneous Upper Confidence Limit

1 - 4	-261.45	-116.90	27.65
1 - 3	-193.28	-89.17	14.95
1 - 2	-181.52	-71.54	38.45
1 - 6	-212.78	-68.23	76.31
1 - 5	-181.08	-63.37	54.34
1 - 8	-84.62	59.93	204.48
1 - 7	5.88	150.43	294.98
8 - 4	-364.42	-176.83	10.76
8 - 3	-307.64	-149.10	9.44
8 - 2	-293.93	-131.47	30.99
8 - 6	-315.76	-128.17	59.42
8 - 5	-291.09	-123.30	44.49
8 - 1	-204.48	-59.93	84.62
8 - 7	-97.09	90.50	278.09
7 - 4	-454.92	-267.33	-79.74
7 - 3	-398.14	-239.60	-81.06
7 - 2	-384.43	-221.97	-59.51
7 - 6	-406.26	-218.67	-31.08
7 - 5	-381.59	-213.80	-46.01
7 - 1	-294.98	-150.43	-5.88
7 - 8	-278.09	-90.50	97.09

SAMPLE=B1

SAMPLE=B1

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSF= 286.634
Critical Value of Studentized Range= 4.421

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous Lower Confidence Limit		Difference Between Means	Simultaneous Upper Confidence Limit	
5	- 2	-34.155		-3.984	26.188	***
5	- 7	146.149		184.800	223.451	***
7	- 4	-348.213		-305.000	-261.787	***
7	- 8	-248.879		-205.667	-162.454	***
7	- 1	-234.480		-200.581	-166.682	***
7	- 3	-229.598		-193.077	-156.556	***
7	- 2	-224.614		-188.784	-152.954	***
7	- 5	-223.451		-184.800	-146.149	***

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means	Simultaneous Upper Confidence Limit	
4 - 8	56.121	99.333	142.546	***	
4 - 1	70.520	104.419	138.318	***	
4 - 3	75.402	111.923	148.444	***	
4 - 2	80.386	116.216	152.046	***	
4 - 5	81.549	120.200	158.851	***	
4 - 7	261.787	305.000	348.213	***	
8 - 4	-142.546	-99.333	-56.121	***	
8 - 1	-28.813	5.086	38.985		
8 - 3	-23.932	12.590	49.111		
8 - 2	-18.947	16.883	52.713		
8 - 5	-17.784	20.867	59.517	***	
8 - 7	162.454	205.667	248.879	***	
1 - 4	-138.318	-104.419	-70.520	***	
1 - 8	-38.985	-5.086	28.813		
1 - 3	-17.308	7.504	32.315		
1 - 2	-11.985	11.797	35.579		
1 - 5	-12.070	15.781	43.631	***	
1 - 7	166.682	200.581	234.480	***	
3 - 4	-148.444	-111.923	-75.402	***	
3 - 8	-49.111	-12.590	23.932		
3 - 1	-32.315	-7.504	17.308		
3 - 2	-23.098	4.293	31.684		
3 - 5	-22.712	8.277	39.267	***	
3 - 7	156.556	193.077	229.598	***	
2 - 4	-152.046	-116.216	-80.386	***	
2 - 8	-52.713	-16.883	18.947		
2 - 1	-35.579	-11.797	11.985		
2 - 3	-31.684	-4.293	23.098		
2 - 5	-26.188	3.984	34.155	***	
2 - 7	152.954	188.784	224.614	***	
5 - 4	-158.851	-120.200	-81.549	***	
5 - 8	-59.517	-20.867	17.784		
5 - 1	-43.631	-15.781	12.070		
5 - 3	-39.267	-8.277	22.712		

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Mn

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 35 MSE= 12.99239
Critical Value of Studentized Range= 4.421

Number of observations in by group = 42

Comparisons significant at the 0.05 level are indicated by '****'.

Dependent Variable: Mn				
Source	DF	Sum of Squares	F Value	Pr > F
Model	6	5439.65607052	69.78	0.0001
Error	35	454.73351995		
Corrected Total	41	5894.38959048		
R-Square		C.V.	Mn Mean	
	0.922853	25.81312	13.96380952	
Type I SS				
Source	DF	Type I SS	F Value	Pr > F
METH	6	5439.65607052	69.78	0.0001
Type III SS				
Source	DF	Type III SS	F Value	Pr > F
METH	6	5439.65607052	69.78	0.0001

METH Comparison		Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit			
4	- 8	8.410	17.610	26.810	***	
4	- 1	29.623	36.841	44.058	***	
4	- 5	31.382	39.611	47.839	***	
4	- 2	32.082	39.710	47.339	***	
4	- 3	34.185	41.961	49.736	***	
4	- 7	33.767	42.967	52.167	***	
8	- 4	-26.810	-17.610	-8.410	***	
8	- 1	12.013	19.231	26.448	***	
8	- 5	13.772	22.001	30.229	***	
8	- 2	14.472	22.100	29.729	***	
8	- 3	16.575	24.351	32.126	***	
8	- 7	16.157	25.357	34.557	***	
1	- 4	-44.058	-36.841	-29.623	***	
1	- 8	-26.448	-19.231	-12.013	***	
1	- 5	-3.159	2.770	8.700		
1	- 2	-2.193	2.870	7.933		
1	- 3	-0.162	5.120	10.403		
1	- 7	-1.091	6.126	13.343		
5	- 4	-47.839	-39.611	-31.382	***	
5	- 8	-30.229	-22.001	-13.772	***	
5	- 1	-8.700	-2.770	3.159		
5	- 2	-6.324	0.100	6.523		
5	- 3	-4.247	2.350	8.948		
5	- 7	-4.873	3.356	11.585		
2	- 4	-47.339	-39.710	-32.082	***	
2	- 8	-29.729	-22.100	-14.472	***	
2	- 1	-7.933	-2.870	2.193		
2	- 5	-6.523	-0.100	6.324		
2	- 3	-3.581	2.251	8.082		
2	- 7	-4.372	3.256	10.885		
3	- 4	-49.736	-41.961	-34.185	***	
3	- 8	-32.126	-24.351	-16.575	***	
3	- 1	-10.403	-5.120	0.162		
3	- 5	-8.948	-2.350	4.247		

SAMPLE=B2

General Linear Models Procedure

METH Comparison	Simultaneous		Difference		Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Lower Confidence Limit	Upper Confidence Limit
3 - 2	-8.082	3.581	-2.251	3.581		
3 - 7	-6.770	8.781	1.006	8.781		
7 - 4	-52.167	-33.767	-42.967	-33.767	***	
7 - 8	-34.557	-16.157	-25.357	-16.157	***	
7 - 1	-13.343	1.091	-6.126	1.091		
7 - 5	-11.585	4.873	-3.356	4.873		
7 - 2	-10.885	4.372	-3.256	4.372		
7 - 3	-8.781	6.770	-1.006	6.770		

33. Cr

units: mg/kg
optional for all samples
reported by 9 laboratories

TABLE I : Cr - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1			
2			
3	A1 A2	5	N
4	A2	4	N
5			
6			
7	A2	3	N
8	A2	3	N
9	A1 A2 B1 B2	3	N
10	A2	3	R
11			
12			
13			
14			
15	A2	3	N
16			
17			
18	A2	3	N/R
19	A2	3	R
20			
21			
22			

(*) R = aqua regia

TABLE II : Cr - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HClO ₄ + HNO ₃
3	total analysis, 2M HNO ₃
4	AFNOR X31-151
5	minéralisation

TABLE V :

----- SAMPLE=A2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cr

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 22 MSE= 156.9541
Critical Value of Studentized Range= 4.196

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison		Simultaneous		Difference		Simultaneous	
		Lower	Upper	Between		Confidence	Limit
		Confidence	Limit	Means		Limit	
4	- 1	-19.835	5.815			31.466	
4	- 5	-21.140	9.210			39.560	
4	- 3	-8.465	17.185			42.836	
4	- 2	-4.158	21.492			47.143	
1	- 4	-31.466	-5.815			19.835	
1	- 5	-22.256	3.395			29.045	
1	- 3	-8.499	11.370			31.239	
1	- 2	-4.192	15.677			35.546	
5	- 4	-39.560	-9.210			21.140	
5	- 1	-29.045	-3.395			22.256	
5	- 3	-17.675	7.975			33.626	
5	- 2	-13.368	12.282			37.933	
3	- 4	-42.836	-17.185			8.465	
3	- 1	-31.239	-11.370			8.499	
3	- 5	-33.626	-7.975			17.675	
3	- 2	-15.562	4.307			24.176	
2	- 4	-47.143	-21.492			4.158	
2	- 1	-35.546	-15.677			4.192	
2	- 5	-37.933	-12.282			13.368	
2	- 3	-24.176	-4.307			15.562	

TABLE III : Cr - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	26.45	27.32	28.43	0.67	3.55
9	2	3	23.00	26.33	29.00	3.06	11.60

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	16.65	18.48	20.43	1.55	8.39
4	3	4	6.15	6.29	6.51	0.18	2.79
5							
6							
7	4	3	32.90	35.07	37.50	2.31	6.59
8	5	3	24.77	25.86	27.02	1.13	4.36
9	2	3	11.00	12.33	14.00	1.53	12.39
10	1	3	13.00	13.50	14.00	0.50	3.70
11							
12							
13							
14							
15	2	3	11.76	13.18	14.07	1.24	9.43
16							
17							
18	1	3	47.61	49.42	50.76	1.63	3.29
19	3	3	32.39	33.33	34.78	1.27	3.82
20	1	3		16.00			
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	35.41	36.78	38.37	1.06	2.88
9	2	3	26.00	28.67	31.00	2.52	8.78

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	7.39	8.19	9.24	0.71	8.67
9	2	3	4.00	4.33	5.00	0.58	13.32

TABLE IV : Cr - Total mean and normal distribution

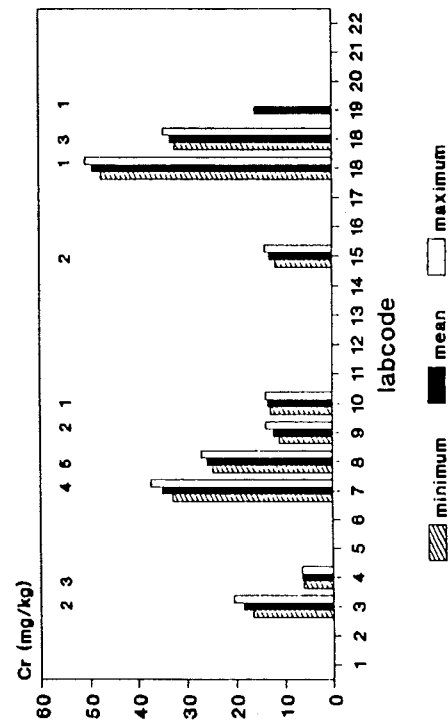
sample	method	repl	mean	std	normal	prob<W
A1	2	4	26.58	2.54	0.90	1.00
A2	1	7	29.25	18.91	0.72	0.01
A2	2	7	13.57	2.48	0.86	0.17
A2	3	7	17.88	14.47	0.69	0.00
A2	4	3	35.07	2.31	0.99	0.81
A2	5	3	25.86	1.13	1.00	0.89
B1	2	4	30.70	4.55	0.97	0.79
B2	2	4	5.30	1.99	0.79	0.08

Remark:

Labcode 3 and labcode 19 reported mean values and the number of replicates, respectively 5 and 3.

To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.

So, the "repl" values in this table can sometimes be different from the real number of replicates when these 2 laboratories are included.

Cr sample A2
national/reference method

34. Ni

units: mg/kg
optional for sample A2
reported by 8 laboratories

TABLE I : Ni - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
1			
2			
3	A1 A2 B1 B2	5	N
4	A2	4	N
5			
6			
7	A2	3	N
8	A2	3	N
9	A1 A2 B1 B2	3	N
10			
11			
12			
13			
14			
15	A2	2	N
16			
17			
18	A2	3	N/R
19	A2	3	R
20			
21			
22			

(*) R = aqua regia

TABLE II : Ni - Summary method descriptions

method	description
1	aqua regia
2	total ICP-AES, HClO4 + HNO3
3	total analysis, 2M HNO3
4	AFNOR X31-151
5	minéralisation

TABLE III : Ni - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	27.60	28.34	29.36	0.76	2.68
9	2	3	38.00	39.00	40.00	1.00	2.56

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5	13.89	15.28	15.28	0.85	5.56
4	3	4	5.18	5.64	6.32	0.49	8.63
5							
6							
7	4	3	22.00	23.53	24.40	1.33	5.66
8	5	3	78.40	80.04	83.25	2.78	3.47
9	2	3	16.00	18.00	20.00	2.00	11.11
10							
11							
12							
13							
14							
15	2	2	10.47	11.32	12.16	1.20	10.56
16							
17							
18	1	3	11.96	12.65	13.26	0.65	5.16
19	3	3	10.65	10.83	11.09	0.23	2.11
20	1	3		14.00			
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	20.56	21.21	22.18	0.57	2.69
9	2	3	25.00	26.67	29.00	2.08	7.81

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
3	2	5	1.17	1.92	3.31	0.75	39.06
9	2	3	4.00	4.00	4.00	0.00	0.00

TABLE IV : Ni - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	2	4	36.34	5.39	0.77	0.05
A2	1	4	12.99	0.86	1.00	0.00
A2	2	6	15.32	3.55	0.97	0.90
A2	3	7	7.86	2.80	0.76	0.02
A2	4	3	23.53	1.33	0.81	0.14
A2	5	3	80.04	2.78	0.76	0.02
B1	2	4	25.30	3.21	0.98	0.90
B2	2	4	3.48	1.04	0.63	0.00

Remark:

Labcode 3 and labcode 19 reported mean values and the number of replicates, respectively 5 and 3.

To calculate the total number of replicates for the method-grouped laboratories, SAS counts the number of reported results.

So, the "repl" values in this table for methods 1 and 2 are different from the real number of replicates.

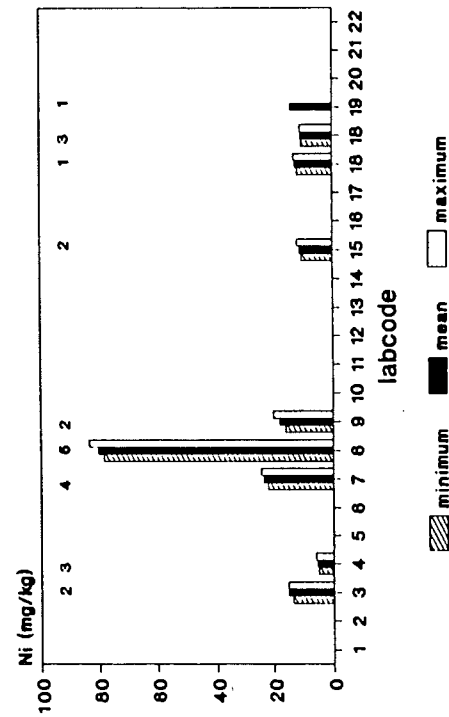
Ni sample A2
national method

TABLE V : Ni Method comparison 644
 ----- SAMPLE=A2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Ni

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 18 MSE= 7.301566
 Critical Value of Studentized Range= 4.276

Comparisons significant at the 0.05 level are indicated by '****'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
5	- 4	49.835	56.507	63.178	***	***
5	- 2	58.944	64.722	70.499	***	***
5	- 1	60.815	67.055	73.295	***	***
5	- 3	66.539	72.177	77.815	***	***
4	- 5	-63.178	-56.507	-49.835	***	***
4	- 2	2.437	8.215	13.993	***	***
4	- 1	4.308	10.548	16.789	***	***
4	- 3	10.032	15.670	21.309	***	***
2	- 5	-70.499	-64.722	-58.944	***	***
2	- 4	-13.993	-8.215	-2.437	***	***
2	- 1	-2.941	2.333	7.608	***	***
2	- 3	2.910	7.455	12.001	***	***
1	- 5	-73.295	-67.055	-60.815	***	***
1	- 4	-16.789	-10.548	-4.308	***	***
1	- 2	-7.608	-2.333	2.941	***	***
1	- 3	0.001	5.122	10.243	***	***
3	- 5	-77.815	-72.177	-66.539	***	***
3	- 4	-21.309	-15.670	-10.032	***	***
3	- 2	-12.001	-7.455	-2.910	***	***
3	- 1	-10.243	-5.122	-0.001	***	***

General Linear Models Procedure
 Class Level Information

Class	Levels	Values
METH	5	1 2 3 4 5

Number of observations in by group = 23

Dependent Variable: Ni

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	12104.3720941	414.44	0.0001
Error	18	131.4281929		
Corrected Total	22	12235.8002870		
R-Square		C.V.	Ni Mean	
0.989259		12.19545	22.15695652	

Source	DF	Type I SS	F Value	Pr > F
METH	4	12104.3720941	414.44	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	4	12104.3720941	414.44	0.0001

35. Cd

units: mg/kg
optional for all samples
reported by 10 laboratories

TABLE I : Cd - Overview of reported data

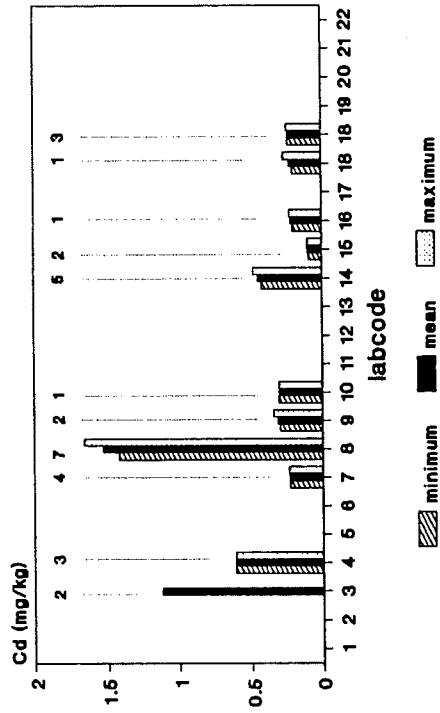
labcode	samples reported	number of replicates	N/R method (*)
1			
2	A1 A2 B1 B2	5	N
3	A1 A2 B1 B2	4	N
4			
5			
6			
7	A1 A2 B1 B2	3	N
8	A1 A2 B1 B2	3	N
9	A1 A2 B1 B2	3	N
10	A1 A2 B1 B2	3	N=R
11			
12			
13			
14	A1 A2 A3 B1 B2	4	N
15	A1 A2 B1 B2	A1 B1:1 A2:2 B2:4	N
16	A1 A2 B1 B2	3	N=R
17			
18	A1 A2 B1 B2	3	N/R
19			
20			
21			
22			

(*) R = aqua regia

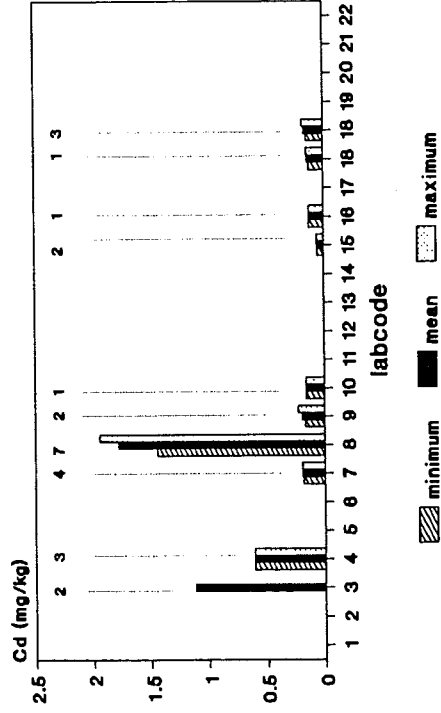
TABLE II : Cd - Summary method descriptions

method	description
1	aqua regia
2	HClO4 + HNO3
3	2M HNO3
4	AFNOR X31-151
5	dry ashing, HCl
6	digestion, HNO3 under pressure
7	minéralisation

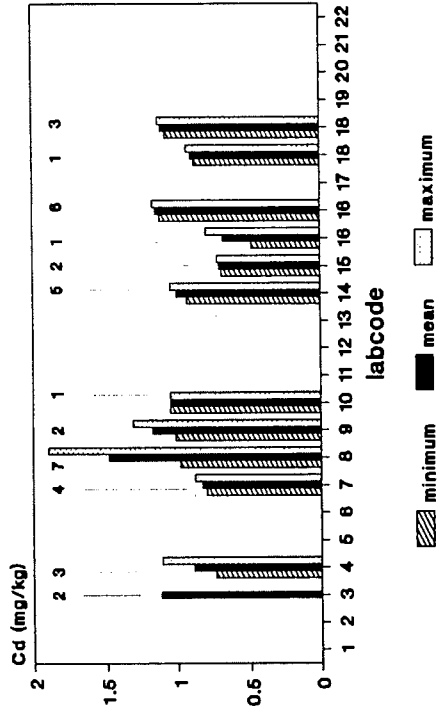
Cd sample A1
national methods



Cd sample B1
national methods



Cd sample A2
national methods



Cd sample B2
national methods

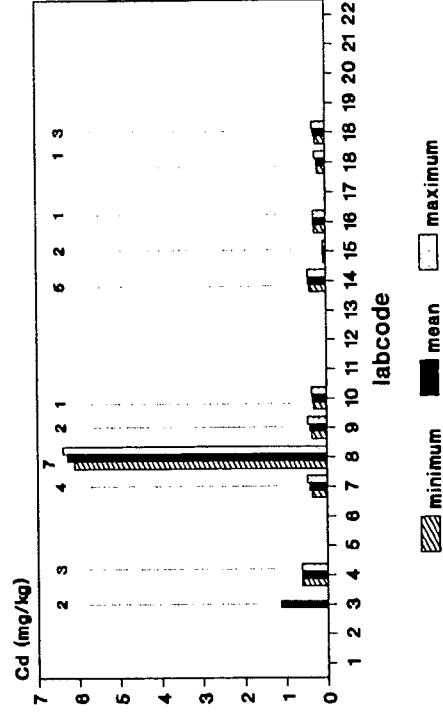


TABLE III : Cd - Descriptive statistics

sample A1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5		1.12			
4	3	4	0.61	0.61	0.61	0.00	0.00
5							
6							
7	4	3	0.22	0.22	0.23	0.01	2.59
8	7	3	1.42	1.53	1.66	0.12	8.00
9	2	3	0.29	0.31	0.34	0.03	8.54
10	1	3	0.30	0.30	0.30	0.00	0.00
11							
12							
13							
14	5	4	0.42	0.45	0.48	0.03	5.74
15	2	3	0.09	0.10	0.10	0.01	5.97
16	1	3	0.20	0.21	0.22	0.01	4.76
17							
18	1	3	0.20	0.22	0.26	0.04	15.75
18	3	3	0.23	0.23	0.24	0.01	2.47
19							
20							
21							
22							

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5		1.12			
4	3	4	0.74	0.89	1.11	0.17	19.17
5							
6							
7	4	3	0.80	0.83	0.88	0.04	5.00
8	7	3	0.98	1.48	1.90	0.47	31.42
9	2	3	1.01	1.18	1.31	0.15	12.98
10	1	3	1.05	1.05	1.05	0.00	0.00
11							
12							
13							
14	5	5	0.93	1.01	1.05	0.05	4.53
14	A3	5	1.03	1.04	1.06	0.02	1.45
15	2	2	0.69	0.71	0.72	0.02	3.01
16	1	3	0.48	0.68	0.80	0.17	25.64
16	6	3	1.12	1.15	1.17	0.03	2.20
17							
18	1	3	0.88	0.90	0.93	0.03	2.79
18	3	3	1.08	1.11	1.13	0.03	2.59
19							
20							
21							
22							

sample B1 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5		1.12			
4	3	4	0.61	0.61	0.61	0.00	0.00
5							
6							
7	4	3	0.18	0.19	0.19	0.01	3.09
8	7	3	1.45	1.78	1.94	0.28	15.92
9	2	3	0.16	0.19	0.22	0.03	15.79
10	1	3	0.15	0.15	0.15	0.00	0.00
11							
12							
13							
14	5	5		0.00			
15	2	3	0.05	0.06	0.06	0.01	10.19
16	1	3	0.12	0.12	0.12	0.00	0.00
17							
18	3	3	0.14	0.16	0.18	0.02	12.75
18	1	3	0.12	0.13	0.14	0.01	8.66
19							
20							
21							
22							

sample B2 - national methods

labcode	method	repl	minimum	mean	maximum	std	cv
1							
2							
3	2	5		1.12			
4	3	4	0.61	0.61	0.61	0.00	0.00
5							
6							
7	4	3	0.34	0.40	0.46	0.06	15.00
8	7	3	6.10	6.27	6.37	0.15	2.40
9	2	3	0.34	0.40	0.45	0.06	13.89
10	1	3	0.30	0.33	0.35	0.03	8.66
11							
12							
13							
14	5	5	0.39	0.42	0.43	0.02	4.02
15	2	4	0.05	0.06	0.07	0.01	15.32
16	1	3	0.26	0.28	0.29	0.02	6.19
17							
18	1	3	0.17	0.19	0.24	0.04	20.90
18	3	3	0.22	0.26	0.30	0.04	15.35
19							
20							
21							
22							

SAMPLE=A2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	7	1 2 3 4 5 6 7

Number of observations in by group = 36

Dependent Variable: Cd

Source	DF	Sum of Squares	F Value	Pr > F
Model	6	0.98362802	3.89	0.0057
Error	29	1.22244698		
Corrected Total	35	2.20607500		
R-Square				
C.V.				
Cd Mean				
		20.37844		1.00750000
Type I SS				
Source	DF	F Value	Pr > F	
METH	6	0.98362802	3.89	0.0057
Type III SS				
Source	DF	F Value	Pr > F	
METH	6	0.98362802	3.89	0.0057

SAMPLE=A1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 27 MSE= 0.040089
Critical Value of Studentized Range= 4.333

Comparisons significant at the 0.05 level are indicated by '****'.

METH Comparison	Simultaneous Lower Confidence Limit		Difference Between Means		Simultaneous Upper Confidence Limit		
7 - 5	0.6081	1.0767	1.5452	***			
7 - 3	0.6548	1.0781	1.5014	***			
7 - 2	0.7691	1.1924	1.6157	***			
7 - 1	0.8744	1.2833	1.6923	***			
7 - 4	0.8024	1.3033	1.8042	***			
5 - 7	-1.5452	-1.0767	-0.6081	***			
5 - 3	-0.3831	0.0014	0.3859				
5 - 2	-0.2688	0.1157	0.5002				
5 - 1	-0.1620	0.2067	0.5753				
5 - 4	-0.2419	0.2267	0.6952				
3 - 7	-1.5014	-1.0781	-0.6548	***			
3 - 5	-0.3859	-0.0014	0.3831				
3 - 2	-0.2136	0.1143	0.4422				
3 - 1	-0.1039	0.2052	0.5144				
3 - 4	-0.1981	0.2252	0.6486				
2 - 7	-1.6157	-1.1924	-0.7691	***			
2 - 5	-0.5002	-0.1157	0.2688				
2 - 3	-0.4422	-0.1143	0.2136				
2 - 1	-0.2182	0.0910	0.4001				
2 - 4	-0.3124	0.1110	0.5343				
1 - 7	-1.6923	-1.2833	-0.8744	***			
1 - 5	-0.5753	-0.2067	0.1620				
1 - 3	-0.5144	-0.2052	0.1039				
1 - 2	-0.4001	-0.0910	0.2182				
1 - 4	-0.3890	0.0200	0.4290				
4 - 7	-1.8042	-1.3033	-0.8024	***			
4 - 5	-0.6952	-0.2267	0.2419				
4 - 3	-0.6486	-0.2252	0.1981				
4 - 2	-0.5343	-0.1110	0.3124				
4 - 1	-0.4290	-0.0200	0.3890				

TABLE V : Cd method comparison

SAMPLE=A1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 7

Number of observations in by group = 33

Dependent Variable: Cd

Source	DF	Sum of Squares	F Value	Pr > F
Model	5	4.10939740	20.50	0.0001
Error	27	1.08239048		
Corrected Total	32	5.19178788		

R-Square C.V. Cd Mean

0.791519 44.88655 0.44606061

Source	DF	Type I SS	F Value	Pr > F
METH	5	4.10939740	20.50	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	5	4.10939740	20.50	0.0001

TABLE IV : Cd - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob<W
A1	1	9	0.24	0.05	0.79	0.01
A1	2	7	0.33	0.36	0.70	0.00
A1	3	7	0.45	0.20	0.67	0.00
A1	4	3	0.22	0.01	0.75	0.00
A1	5	4	0.45	0.03	0.99	0.96
A1	7	3	1.53	0.12	0.96	0.64
A2	1	9	0.88	0.18	0.87	0.11
A2	2	6	1.01	0.26	0.91	0.40
A2	3	7	0.98	0.17	0.81	0.05
A2	4	8	0.83	0.04	0.92	0.46
A2	5	5	1.01	0.05	0.86	0.22
A2	6	3	1.15	0.03	0.99	0.78
A2	7	3	1.48	0.47	0.97	0.69
A3	4	5	1.04	0.02	0.80	0.08
B1	1	9	0.13	0.01	0.77	0.01
B1	2	7	0.27	0.38	0.62	0.00
B1	3	7	0.42	0.24	0.69	0.00
B1	4	3	0.19	0.01	0.75	0.00
B1	7	3	1.78	0.28	0.75	0.00
B2	1	9	0.27	0.07	0.91	0.29
B2	2	8	0.32	0.36	0.77	0.01
B2	3	7	0.46	0.19	0.72	0.01
B2	4	3	0.40	0.06	1.00	0.99
B2	5	5	0.42	0.02	0.88	0.31
B2	7	3	6.27	0.15	0.81	0.13

Remark:

Labcode 3 reported mean values for 5 replicates.

To calculate the total number of replicates for the method-grouped laboratories,

SAS counts the number of reported results.

So, the "repl" values in this table for method 2 are different from the real number of replicates.

SAMPLE=A2

SAMPLE=A2

General Linear Models Procedure

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 29 MSE= 0.042153
Critical Value of Studentized Range= 4.475

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Upper Confidence Limit
1 - 3	-0.4339		-0.1065		0.2209
1 - 4	-0.3886		0.0444		0.4775
4 - 7	-1.1804		-0.6500		-0.1196 ***
4 - 6	-0.8438		-0.3133		0.2171
4 - 2	-0.6360		-0.1767		0.2827
4 - 5	-0.6471		-0.1727		0.3018
4 - 3	-0.5992		-0.1510		0.2973
4 - 1	-0.4775		-0.0444		0.3886

METH Comparison	Simultaneous		Difference		Simultaneous
	Lower Confidence Limit	Upper Confidence Limit	Between Means	Upper Confidence Limit	Upper Confidence Limit
7 - 6	-0.1938		0.3367		0.8671 ***
7 - 2	0.0140		0.4733		0.9327 ***
7 - 5	0.0029		0.4773		0.9518 ***
7 - 3	0.0508		0.4990		0.9473 ***
7 - 1	0.1725		0.6056		1.0386 ***
7 - 4	0.1196		0.6500		1.1804 ***
6 - 7	-0.8671		-0.3367		0.1938
6 - 2	-0.3227		0.1367		0.5960
6 - 5	-0.3338		0.1407		0.6151
6 - 3	-0.2859		0.1624		0.6107
6 - 1	-0.1642		0.2689		0.7020
6 - 4	-0.2171		0.3133		0.8438
2 - 7	-0.9327		-0.4733		-0.0140 ***
2 - 6	-0.5960		-0.1367		0.3227
2 - 5	-0.3894		0.0040		0.3974
2 - 3	-0.3357		0.0257		0.3871
2 - 1	-0.2102		0.1322		0.4746
2 - 4	-0.2827		0.1767		0.6360
5 - 7	-0.9518		-0.4773		-0.0029 ***
5 - 6	-0.6151		-0.1407		0.3338
5 - 2	-0.3974		-0.0040		0.3894
5 - 3	-0.3587		0.0217		0.4021
5 - 1	-0.2341		0.1282		0.4906
5 - 4	-0.3018		0.1727		0.6471
3 - 7	-0.9473		-0.4990		-0.0508 ***
3 - 6	-0.6107		-0.1624		0.2859
3 - 2	-0.3871		-0.0257		0.3357
3 - 5	-0.4021		-0.0217		0.3587
3 - 1	-0.2209		0.1065		0.4339
3 - 4	-0.2973		0.1510		0.5992
1 - 7	-1.0386		-0.6056		-0.1725 ***
1 - 6	-0.7020		-0.2689		0.1642
1 - 2	-0.4746		-0.1322		0.2102
1 - 5	-0.4906		-0.1282		0.2341

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 24 MSE= 0.057692
Critical Value of Studentized Range= 4.166

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous		Difference Between Means	Simultaneous	
	Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 3	0.870	1.358	1.358	0.870	1.846
7 - 2	1.023	1.511	1.511	1.023	1.999
7 - 4	1.012	1.590	1.590	1.012	2.168
7 - 1	1.170	1.642	1.642	1.170	2.114
3 - 7	-1.846	-1.358	-1.358	-1.846	-0.870
3 - 2	-0.225	0.153	0.153	-0.225	0.531
3 - 4	-0.256	0.232	0.232	-0.256	0.720
3 - 1	-0.072	0.284	0.284	-0.072	0.641
2 - 7	-1.999	-1.511	-1.511	-1.999	-1.023
2 - 3	-0.531	-0.153	-0.153	-0.531	0.225
2 - 4	-0.409	0.079	0.079	-0.409	0.567
2 - 1	-0.225	0.131	0.131	-0.225	0.488
4 - 7	-2.168	-1.590	-1.590	-2.168	-1.012
4 - 3	-0.720	-0.232	-0.232	-0.720	0.256
4 - 2	-0.567	-0.079	-0.079	-0.567	0.409
4 - 1	-0.420	0.052	0.052	-0.420	0.524
1 - 7	-2.114	-1.642	-1.642	-2.114	-1.170
1 - 3	-0.641	-0.284	-0.284	-0.641	0.072
1 - 2	-0.488	-0.131	-0.131	-0.488	0.225
1 - 4	-0.524	-0.052	-0.052	-0.524	0.420

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 7

Number of observations in by group = 30

NOTE: Due to missing values, only 29 observations can be used in this analysis.

Dependent Variable: Cd

Source	DF	Sum of Squares	F Value	Pr > F
Model	4	6.58258730	28.52	0.0001
Error	24	1.38461270		
Corrected Total	28	7.96720000		

R-Square C.V. Cd Mean

0.826211 58.58341 0.41000000

Source	DF	Type I SS	F Value	Pr > F
METH	4	6.58258730	28.52	0.0001
Source	DF	Type III SS	F Value	Pr > F
METH	4	6.58258730	28.52	0.0001

----- SAMPLE=A2 -----

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 29 MSE= 0.042153
Critical Value of Studentized Range= 4.475

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
7 - 6	-0.1938	0.3367	0.8671	0.8671	0.3367	0.8671
7 - 2	0.0140	0.4733	0.9327	0.9327	0.4733	0.9327
7 - 5	0.0029	0.4773	0.9518	0.9518	0.4773	0.9518
7 - 3	0.0508	0.4990	0.9473	0.9473	0.4990	0.9473
7 - 1	0.1725	0.6056	1.0386	1.0386	0.6056	1.0386
7 - 4	0.1196	0.6500	1.1804	1.1804	0.6500	1.1804
6 - 7	-0.8671	-0.3367	0.1938	0.1938	-0.3367	0.8671
6 - 2	-0.3227	0.1367	0.5960	0.5960	0.1367	1.1804
6 - 5	-0.3338	0.1407	0.6151	0.6151	0.1407	1.1804
6 - 3	-0.2859	0.1624	0.6107	0.6107	0.1624	1.1804
6 - 1	-0.1642	0.2689	0.7020	0.7020	0.2689	1.1804
6 - 4	-0.2171	0.3133	0.8438	0.8438	0.3133	1.1804
2 - 7	-0.9327	-0.4733	-0.0140	-0.0140	-0.4733	-0.9327
2 - 6	-0.5960	-0.1367	0.3227	0.3227	-0.1367	0.8671
2 - 5	-0.3894	0.0040	0.3974	0.3974	0.0040	0.8671
2 - 3	-0.3357	0.0257	0.3871	0.3871	0.0257	0.8671
2 - 1	-0.2102	0.1322	0.4746	0.4746	0.1322	0.8671
2 - 4	-0.2827	0.1767	0.6360	0.6360	0.1767	0.8671
5 - 7	-0.9518	-0.4773	-0.0029	-0.0029	-0.4773	-0.9518
5 - 6	-0.6151	-0.1407	0.3338	0.3338	-0.1407	0.8671
5 - 2	-0.3974	-0.0040	0.3894	0.3894	-0.0040	0.8671
5 - 3	-0.3587	0.0217	0.4021	0.4021	0.0217	0.8671
5 - 1	-0.2341	0.1282	0.4906	0.4906	0.1282	0.8671
5 - 4	-0.3018	0.1727	0.6471	0.6471	0.1727	0.8671
3 - 7	-0.9473	-0.4990	-0.0508	-0.0508	-0.4990	-0.9473
3 - 6	-0.6107	-0.1624	0.2859	0.2859	-0.1624	0.8671
3 - 2	-0.3871	-0.0257	0.3357	0.3357	-0.0257	0.8671
3 - 5	-0.4021	-0.0217	0.3587	0.3587	-0.0217	0.8671
3 - 1	-0.2209	0.1065	0.4339	0.4339	0.1065	0.8671
3 - 4	-0.2973	0.1510	0.5992	0.5992	0.1510	0.8671
1 - 7	-1.0386	-0.6056	-0.1725	-0.1725	-0.6056	-1.0386
1 - 6	-0.7020	-0.2689	0.1642	0.1642	-0.2689	0.8671
1 - 2	-0.4746	-0.1322	0.2102	0.2102	-0.1322	0.8671
1 - 5	-0.4906	-0.1282	0.2341	0.2341	-0.1282	0.8671

----- SAMPLE=A2 -----

General Linear Models Procedure

Simultaneous

Lower Confidence Limit

Difference Between Means

Upper Confidence Limit

METH Comparison	Simultaneous			Simultaneous		
	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit	Lower Confidence Limit	Difference Between Means	Upper Confidence Limit
1 - 3	-0.4339	-0.1065	0.2209	0.2209	-0.1065	0.4775
1 - 4	-0.3886	0.0444	0.4775	0.4775	0.0444	0.9219
4 - 7	-1.1804	-0.6500	-0.1196	-0.1196	-0.6500	-0.6500
4 - 6	-0.8438	-0.3133	0.2171	0.2171	-0.3133	0.7274
4 - 2	-0.6360	-0.1767	0.2827	0.2827	-0.1767	0.7594
4 - 5	-0.6471	-0.1727	0.3018	0.3018	-0.1727	0.7745
4 - 3	-0.5992	-0.1510	0.2973	0.2973	-0.1510	0.7463
4 - 1	-0.4775	-0.0444	0.3886	0.3886	-0.0444	0.8330

SAMPLE=B1

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 24 MSE= 0.057692
Critical Value of Studentized Range= 4.166

Comparisons significant at the 0.05 level are indicated by '***'.

METH Comparison		Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7 - 3		0.870	1.358	1.358	1.846	***
7 - 2		1.023	1.511	1.511	1.999	***
7 - 4		1.012	1.590	1.590	2.168	***
7 - 1		1.170	1.642	1.642	2.114	***
3 - 7		-1.846	-1.358	-1.358	-0.870	***
3 - 2		-0.225	0.153	0.153	0.531	
3 - 4		-0.256	0.232	0.232	0.720	
3 - 1		-0.072	0.284	0.284	0.641	
2 - 7		-1.999	-1.511	-1.511	-1.023	***
2 - 3		-0.531	-0.153	-0.153	0.225	
2 - 4		-0.409	0.079	0.079	0.567	
2 - 1		-0.225	0.131	0.131	0.488	
4 - 7		-2.168	-1.590	-1.590	-1.012	***
4 - 3		-0.720	-0.232	-0.232	0.256	
4 - 2		-0.567	-0.079	-0.079	0.409	
4 - 1		-0.420	0.052	0.052	0.524	
1 - 7		-2.114	-1.642	-1.642	-1.170	***
1 - 3		-0.641	-0.284	-0.284	0.072	
1 - 2		-0.488	-0.131	-0.131	0.225	
1 - 4		-0.524	-0.052	-0.052	0.420	

SAMPLE=B1

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 7

Number of observations in by group = 30

NOTE: Due to missing values, only 29 observations can be used in this analysis.

Dependent Variable: Cd				
Source	DF	Sum of Squares	F Value	Pr > F
Model	4	6.58258730	28.52	0.0001
Error	24	1.38461270		
Corrected Total	28	7.96720000		
R-Square C.V. Cd Mean				
		58.58341		0.41000000
Type I SS F Value Pr > F				
Source	DF	Type I SS	F Value	Pr > F
METH	4	6.58258730	28.52	0.0001
Type III SS F Value Pr > F				
Source	DF	Type III SS	F Value	Pr > F
METH	4	6.58258730	28.52	0.0001

SAMPLE=B2

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Cd

NOTE: This test controls the type I experimentwise error rate.

Alpha= 0.05 Confidence= 0.95 df= 29 MSE= 0.04233

Critical Value of Studentized Range= 4.311

Comparisons significant at the 0.05 level are indicated by '***'.

METH	Comparison	Simultaneous		Difference Between Means	Simultaneous	
		Lower Confidence Limit	Upper Confidence Limit		Lower Confidence Limit	Upper Confidence Limit
7	- 3	5.3791	5.8119	5.8119	5.3791	6.2447
7	- 5	5.3993	5.8573	5.8573	5.3993	6.3154
7	- 4	5.3612	5.8733	5.8733	5.3612	6.3855
7	- 2	5.5287	5.9533	5.9533	5.5287	6.3780
7	- 1	5.5863	6.0044	6.0044	5.5863	6.4226
3	- 7	-6.2447	-5.8119	-5.8119	-6.2447	-5.3791
3	- 5	-0.3218	0.0454	0.0454	-0.3218	0.4127
3	- 4	-0.3714	0.0614	0.0614	-0.3714	0.4942
3	- 2	-0.1832	0.1414	0.1414	-0.1832	0.4660
3	- 1	-0.1235	0.1925	0.1925	-0.1235	0.5086
5	- 7	-6.3154	-5.8573	-5.8573	-6.3154	-5.3993
5	- 3	-0.4127	-0.0454	-0.0454	-0.4127	0.3218
5	- 4	-0.4421	0.0160	0.0160	-0.4421	0.4741
5	- 2	-0.2616	0.0960	0.0960	-0.2616	0.4536
5	- 1	-0.2027	0.1471	0.1471	-0.2027	0.4970
4	- 7	-6.3855	-5.8733	-5.8733	-6.3855	-5.3612
4	- 3	-0.4942	-0.0614	-0.0614	-0.4942	0.3714
4	- 5	-0.4741	-0.0160	-0.0160	-0.4741	0.4421
4	- 2	-0.3446	0.0800	0.0800	-0.3446	0.5046
4	- 1	-0.2870	0.1311	0.1311	-0.2870	0.5493
2	- 7	-6.3780	-5.9533	-5.9533	-6.3780	-5.5287
2	- 3	-0.4660	-0.1414	-0.1414	-0.4660	0.1832
2	- 5	-0.4536	-0.0960	-0.0960	-0.4536	0.2616
2	- 4	-0.5046	-0.0800	-0.0800	-0.5046	0.3446
2	- 1	-0.2537	0.0511	0.0511	-0.2537	0.3559
1	- 7	-6.4226	-6.0044	-6.0044	-6.4226	-5.5863
1	- 3	-0.5086	-0.1925	-0.1925	-0.5086	0.1235
1	- 5	-0.4970	-0.1471	-0.1471	-0.4970	0.2027
1	- 4	-0.5493	-0.1311	-0.1311	-0.5493	0.2870
1	- 2	-0.3559	-0.0511	-0.0511	-0.3559	0.2537

SAMPLE=B2

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	6	1 2 3 4 5 7

Number of observations in by group = 35

Dependent Variable: Cd				C.V.		Cd Mean	
Source	DF	Sum of Squares	F Value	Pr > F			
Model	5	96.12127873	454.16	0.0001			
Error	29	1.22756127					
Corrected Total	34	97.34884000					
R-Square				C.V.		Cd Mean	
		23.75772		0.86600000			
Source	DF	Type I SS	F Value	Pr > F			
METH	5	96.12127873	454.16	0.0001			
Source	DF	Type III SS	F Value	Pr > F			
METH	5	96.12127873	454.16	0.0001			

36. Hg

units: mg/kg
optional for sample A2
reported by only 2 laboratories

TABLE I : Hg - Overview of reported data

labcode	samples reported	number of replicates	N/R method (*)
7	A2	3	N
18	A2	3	N/R

TABLE II : Hg - Summary method descriptions

method	description
1	aqua regia
2	2M HNO ₃ , AAS
3	AFNOR X31-151

TABLE III : Hg - Descriptive statistics

sample A2 - national methods

labcode	method	repl	minimum	mean	maximum	std	CV
7	3	3	0.22	0.24	0.26	0.02	8.83
18	1	3	0.37	0.38	0.42	0.09	24.97
18	2	3	0.26	0.28	0.30	0.02	7.57

TABLE IV : Hg - Total mean and normal distribution

sample	method	repl	mean	std	normal	prob < W
A2	1	3	0.39	0.03	0.75	0.00
A2	2	3	0.28	0.02	0.92	0.46
A2	3	3	0.24	0.02	0.92	0.46

Hg sample A2
national methods

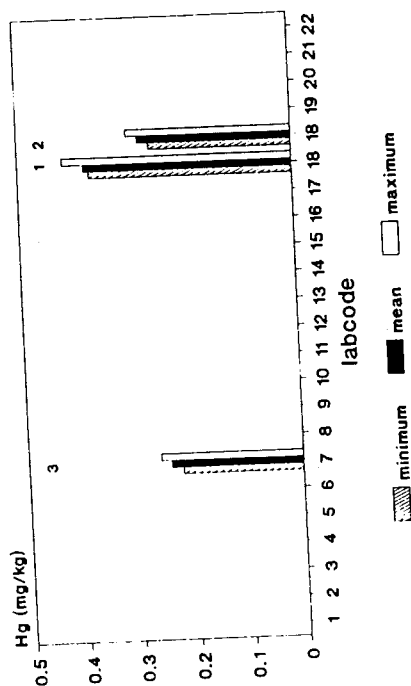


TABLE V : Hg Method comparison

----- SAMPLE=A2 -----

General Linear Models Procedure
Class Level Information

Class	Levels	Values
METH	3	1 2 3

Number of observations in by group = 9

Dependent Variable: Hg

Source	DF	Sum of Squares	F Value	Pr > F
Model	2	0.03282222	28.96	0.0008
Error	6	0.00340000		
Corrected Total	8	0.03622222		
R-Square				
		C.V.		Hg Mean
	0.906135	7.819082		0.30444444

Source	DF	Type I SS	F Value	Pr > F
METH	2	0.03282222	28.96	0.0008
Source	DF	Type III SS	F Value	Pr > F
METH	2	0.03282222	28.96	0.0008

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: Hg

NOTE: This test controls the type I experimentwise error rate,
but generally has a higher type II error rate than REGWQ.Alpha= 0.05 df= 6 MSE= 0.000567
Critical Value of Studentized Range= 4.339
Minimum Significant Difference= 0.0596

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	METH
A	0.3867	3	1
B	0.2833	3	2
B			
B	0.2433	3	3

