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First intercomparison exercise for trace metals in marine biological tissues, NRC BTI/TM

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<https://doi.org/10.4224/8898600>

Report (Marine Analytical Chemistry Standards Program (Canada)), 1985-12

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*Littoral 94
Sept 1987*

Lab 9/87

FIRST INTERCOMPARISON EXERCISE FOR TRACE METALS IN MARINE BIOLOGICAL TISSUES

NRC BT1/TM

Shier Berman and Victor Boyko

REPORT II
NRCC No. 25324

Marine Analytical Chemistry Standards
Standards de chimie analytique marine



National Research Council Conseil national

MARINE ANALYTICAL CHEMISTRY STANDARDS

In 1976, the National Research Council of Canada established a program in support of analytical chemistry aspects of marine science. A recommendation to do so has been made by the Canadian Committee on Oceanography at its 62nd meeting in June, 1975.

In this program, the National Research Council seeks the continuing advice of a Committee on Marine Analytical Chemistry representing chemists active in research on analytical chemistry and of government, university and commercial laboratories applying analytical chemistry in marine science. Further information about this Committee is on the inside back cover of this report.

National Research Council staff at the Atlantic Research Laboratory, Halifax, N. S., and the Division of Chemistry, Ottawa, Ontario, work in support of this program. Much is done in cooperation with other laboratories and through contracts. The results are published in the scientific literature and in this series of reports.

Correspondence should be addressed to Dr. H. A. Gillis, Secretary, Committee on Marine Analytical Chemistry, National Research Council, 1411 Oxford Street, Halifax, N. S., B3H 3Z1. Telephone: (902) 426-9736.

STANDARDS DE CHIMIE ANALYTIQUE MARINE

Le Conseil national de recherches du Canada a créé en 1976 un programme visant à développer certains aspects de la chimie analytique liés aux sciences de la mer. Cette décision donnait suite à une recommandation faite par le Comité canadien d'océanographie lors de sa 62^e réunion en juin 1975.

Dans le cadre de ce programme, le Conseil national de recherches a mis sur pied un Comité permanent de chimie analytique marine représenté par des chimistes, qui effectuent des recherches en chimie analytique, et des laboratoires gouvernementaux, universitaires et commerciaux, qui appliquent la chimie analytique aux sciences de la mer. Vous trouverez de renseignements plus amples au sujet de ce comité à la troisième page de la couverture de ce rapport.

Le personnel du Conseil national de recherches du Laboratoire de recherches de l'Atlantique, situé à Halifax (N.-E.) et celui de la Division de la chimie, situé à Ottawa (Ontario), travaillent sur la réalisation de ce programme. Une bonne partie du travail de recherche se fait en coopération avec d'autres laboratoires et par voie de contrats. Les résultats de ces recherches sont publiés dans les revues scientifiques ainsi que dans les rapports du comité.

Veuillez envoyer le courrier à l'adresse suivante: Dr. H. A. Gillis, Secrétaire du Comité de chimie analytique marine, Conseil national de recherches, 1411, rue Oxford, Halifax (N.-E.) B3H 3Z1. Téléphone: (902) 426-9736.

NATIONAL RESEARCH COUNCIL of CANADA
MARINE ANALYTICAL CHEMISTRY STANDARDS PROGRAM

FIRST INTERCOMPARISON EXERCISE
for
TRACE METALS IN MARINE BIOLOGICAL TISSUES

NRC BT1/TM

by
Shier Berman and Victor Boyko
Marine Analytical Chemistry Standards Program
Division of Chemistry
National Research Council
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December 1985

Abstract

Three marine biological tissue samples (dogfish muscle, scallops, swordfish muscle) were distributed to 51 Canadian and 3 American laboratories, 43 of which submitted results.

Values were collated for the six metals for which all had been asked to analyze: copper, zinc, arsenic, cadmium, mercury and lead. Most laboratories perform well for copper and zinc at the concentrations studied. Performance is also generally good for cadmium and lead where the metal concentrations are respectively greater than a few tenths and several mg/kg of dry tissue.

Less than forty percent of the labs produced satisfactory results for cadmium at the 0.08 mg/kg level in dogfish, and less than twenty-five percent for lead in swordfish at the 0.3 mg/kg level. Less than fifteen percent could cope with the even lower level of lead in the scallops.

A good part of the difficulty lies in the insufficient sensitivity of flame atomic absorption and plasma emission procedures for these metals.

Those that analyze for mercury (less than half the participants) generally do well at levels greater than 1 mg/kg but only a small number did well with the scallops sample at 0.05 mg/kg of mercury.

About half the laboratories submitted results for arsenic with generally good performance.

With but few exceptions, sample preparation techniques appear to be adequate for the six metals in the three samples. Except for low level mercury and cadmium, performance is comparable to recent International Council for the Exploration of the Sea studies.

Results for sodium, calcium, chromium, manganese, iron and nickel are also tabulated.

Résumé

Trois échantillons de tissus biologiques marins (muscle de chien de mer, pétoncle et muscle d'espadon) ont été distribués à 51 laboratoires canadiens et 3 américains, dont 43 ont soumis des résultats.

Des valeurs furent collationnées pour les six métaux que l'on avait demandé d'analyser: le cuivre, le zinc, l'arsenic, le cadmium, le mercure et le plomb. La plupart des laboratoires réussissent bien pour le cuivre et le zinc, aux concentrations étudiées. La réussite est aussi, en général, bonne pour le cadmium et le plomb dont la concentration en métal est supérieure à quelques dizièmes, et plusieurs milligrammes par kilogramme de tissu sec, respectivement.

Moins du quarante pour cent des laboratoires sont arrivés à des résultats satisfaisants pour le cadmium qui se trouve autour de 0.08 mg/kg dans le chien de mer; et moins de vingt-cinq pour cent ont réussi pour le plomb se situant autour de 0.3 mg/kg dans l'espadon. Moins de quinze pour cent ont pu descendre à la concentration de plomb encore plus basse de l'échantillon de pétoncles.

Une bonne partie des difficultés réside dans la sensibilité insuffisante des méthodes d'absorption atomique à flamme et d'émission à plasma pour ces métaux.

Ceux qui déterminent le mercure (moins de la moitié des participants) ont généralement de bons résultats pour des concentrations supérieures à 1 mg/kg, mais seulement un petit nombre a réussi avec l'échantillon de pétoncles possédant une concentration autour de 0.05 mg/kg.

Environ la moitié des laboratoires ont soumis des résultats pour l'arsenic avec, en général, un bon rendement.

Sauf quelques rares exceptions, les techniques de préparation d'échantillon semblent être adéquates pour les six métaux dans les trois échantillons. Mis à part les cas où les concentrations de mercure et de cadmium sont basses, les résultats sont comparables à ceux de récentes études réalisées par le Conseil International pour l'Exploration de la Mer.

On donne également des résultats pour le sodium, le calcium, le chrome, le manganèse, le fer et le nickel.

INTRODUCTION

At its eighth meeting in November 1983, the Committee on Marine Analytical Chemistry, the advisory body to the National Research Council's Marine Analytical Chemistry Standards Program (MACSP), recommended the implementation of two Canadian intercomparison exercises for trace metals: one to be concerned with marine sediments and the other with biological materials. The purpose of these exercises would be to assess the capabilities of Canadian laboratories and make recommendations, where needed, for improvements.

The marine sediment intercomparison, designated as NRC MS1/TM, was carried out in 1984 and a final report has been issued as a MACSP document (Berman and Boyko, 1985).

As opposed to marine sediments where there is a necessity to comply with the provisions of the Ocean Dumping Control Act (ODCA) which controls the dumping of substances harmful to the marine environment, there is no legal compulsion in Canada to make measurements of compositions of marine biological materials. Health and Welfare Canada, within its responsibility to protect Canadians from poisons and harmful substances through the Food and Drugs Act, issued maximum tolerance levels for heavy metals in marine and freshwater animals in 1970. These were revoked in 1979, except for mercury which is at 0.5 mg/kg based on a wet weight of total edible tissue. The identification of areas of increasing deterioration above background is apparently a voluntary responsibility of Fisheries and Oceans Canada.

To our knowledge, there has been no previous large scale Canadian intercomparison exercise regarding trace metals in marine biological materials. The only extensive studies to date have been carried out on an international scale by the International Council for the Exploration of the Sea (ICES) through its Marine Chemistry Working Group, but with little Canadian participation. ICES is currently in the midst of its seventh exercise for trace metals in biological tissue (Berman, 1984). It was hoped that the present study would attract a sufficient number of laboratories across this country so that there would be a good statistical base for conclusions.

PREPARATION OF SAMPLES

Sample D is dogfish muscle material which has been acetone extracted and freeze dried. This sample had been prepared under the direction of Dr. Robin Law in the Fisheries Laboratory, Burnham-on-Crouch, England. The trace metal concentrations in this material were rather higher than originally anticipated but the result was a good sample for this first intercomparison exercise. Samples E and F were obtained from stocks of marine biological tissues prepared at the Technical University of Nova Scotia in Halifax for various NRCC MACSP projects. Sample E is prepared from freshly frozen scallops purchased commercially. The material was thawed, homogenized and spray dried. Sample F is swordfish muscle. The sample, originally in the form of thick steaks, was homogenized and spray dried.

These materials were thoroughly blended and packaged in 18-20 gram quantities in 60-ml bottles. Randomly chosen bottles were analyzed to assess interbottle homogeneity. The materials appear to be relatively homogeneous with respect to copper, zinc, arsenic, cadmium, mercury and lead. The homogeneity is largely reflected in the results of Labs 41A and 41B where replicates were taken from separate bottles.

SAMPLE DISTRIBUTION AND RECEIPT OF RESULTS

The response to a preliminary questionnaire, which promised anonymity to participants, greatly exceeded expectations. Of some 90 laboratories polled, 51 Canadian laboratories indicated

a willingness to participate in this exercise which was designated as NRC MACSP BT1/TM. There was also a request from three National Oceanic and Atmospheric Administration (NOAA) laboratories in the United States for participation.

Sets of samples were sent to these laboratories in August 1984 with the deadline for receipt of results set at December 15. The participants were asked to analyze 6 replicates of each sample for the six metals, copper, zinc, arsenic, cadmium, mercury and lead, and to report the results on a "dry weight" basis, not "as received". Results for other elements were welcome. The laboratories would use their methods of choice. Data regarding the analytical procedures used were requested.

Results were received from about 30 laboratories by mid January, 1985. The Christmas mails did not help the accumulation of results. Receipt of results was finally cut off at the end of February with forty-three laboratories participating. A number was assigned to each of the respondents. Three laboratories submitted more than one set of results for some of the metals.

RESULTS

It is pleasing to note that a good majority of the respondents supplied most of the data requested of them. These included sample drying techniques, decomposition and dissolution procedures, analytical methodologies, calibration techniques and limits of detection for the analyte elements.

All results, except a few very obvious outliers in some sets, were tabulated (pages 26 to 48). The number of significant figures in submitted data was reduced in many cases to a maximum of three for tabulation and computation.

An evaluation has been made for the results for the six core metals of this study (Cu, Zn, As, Cd, Hg and Pb). The data has also been tabulated for those elements where three or more sets of results were received (Na, Ca, Cr, Mn, Fe, and Ni).

A t test at the 95 percent confidence level was applied to the means of all the values submitted (e.g. Liteanu and Rica, 1980). Means were successively rejected (as many as 10 in the case of copper in Sample F) until a normally distributed set of values was obtained. This, of course, implies a normal distribution of results, which may not be a valid assumption. An overall mean, standard deviation and relative standard deviation (RSD) were calculated for the remaining values. This mean and standard deviation are plotted on each graph. The small discrepancy sometimes apparent in the RSD is the result of rounding off significant figures in reporting the mean and standard deviation.

The reason for this approach to the treatment of the data is an attempt to establish reliable consensus values as well as describe the performance of the participants.

An attempt has been made to represent all the data received for the six metals on the individual graphs for each sample:

The range of results from each laboratory (usually 6) and their mean are plotted. Two laboratories submitted two sets of results and one laboratory three sets of results for some of the elements in the samples. These have been plotted sequentially and the term "labs" in this section refers to the number of sets of results, not the number of individual participating laboratories.

A range continued by a forward arrow + beyond the right margin of the graph indicates that the range exceeds this boundary.

A forward arrow → beginning at the right margin indicates that all values submitted exceed the boundary.

A backward arrow ← at the right margin indicates results less than a limit of detection beyond the boundary.

A backward arrow ← within the graph indicates results less than the marked limit of detection.

A backward arrow ← from the left margin indicates results less than the boundary value or a limit of detection which is less than this boundary.

Means marked by a "■" are those that have been retained after the rejection test and are incorporated into the overall result. Means marked by a "+" were rejected.

The results of the various calculations are beside each graph. The total range for all values reported is presented. A mean has been calculated for all quantitative results.

The final means are the consensus values for the concentrations of the six metals in the three samples for this exercise. And, indeed, they may be good estimates of the real concentrations except for mercury and lead in Sample E, where these means may be too high.

The means of the results for copper, zinc, arsenic, cadmium and lead in Samples E and F, and mercury in Samples D and F, have been plotted against each other for those labs that submitted results for both samples. The means and standard deviations indicated on these graphs are those calculated after the rejection of outlying results. This type of plot (Youden, 1968), if the concentrations of the metals are not too different, can indicate those laboratories which consistently produce lower or higher values than the overall mean. Also, those that produce inconsistent results are readily obvious.

Copper

All but four laboratories submitted results for copper. Summaries of the results for this element are listed beside the graphs on page 5 and the overall final means and standard deviations are shown in Table I below. The two numbers in the final column of each table represent the number of quantitative values submitted and values excluded respectively.

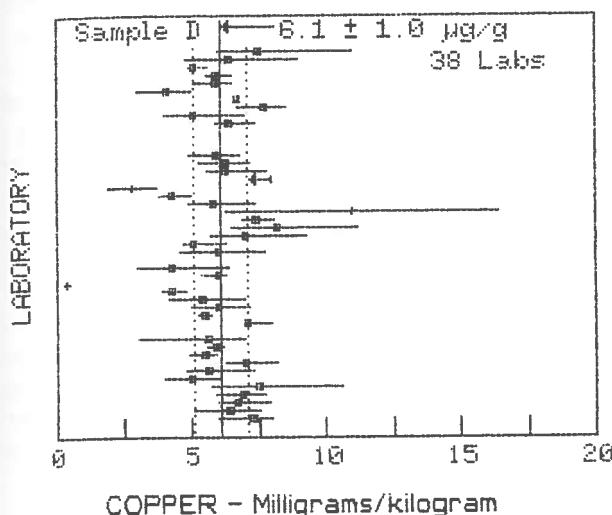
The results for the dogfish sample (D) are quite satisfactory indicating that at least 90 percent of the labs produce acceptable values in this matrix at the 6 mg/kg level. At the concentration range of 1 and 3 mg/kg in Samples E and F respectively the performance deteriorates with only 65 to 70 percent of the labs within the accepted group, but the interlab precision of this group is good. The scallops matrix (E) appears to present the greatest difficulty. The copper concentration is not that much lower than in the swordfish (F) but there is a significantly greater spread in results.

Table I

Copper Concentrations - mg/kg

Sample D	6.1 ± 1.0 (17%)	41, 3
Sample E	1.2 ± 0.2 (20%)	35, 8
Sample F	2.6 ± 0.3 (13%)	39, 10

COPPER DATA

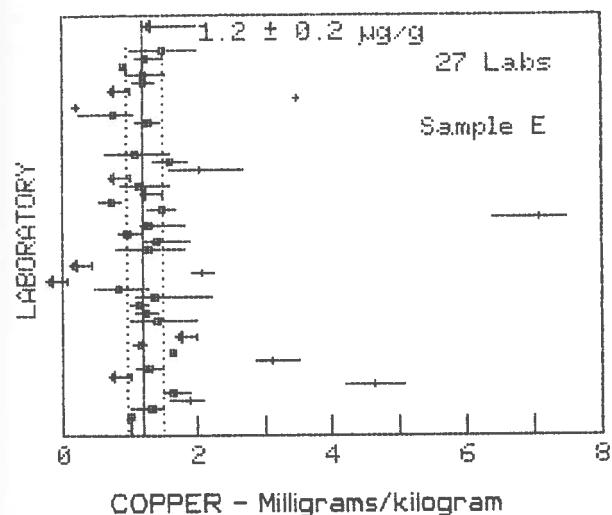


42 labs submitted results ranging from 0.3 to 16.4 mg/kg.

41 labs submitted quantitative values with a range of 0.3 to 16.4 and a mean of $6.0 \pm 1.6 \text{ mg/kg}$.

A *t* test rejected the results of 3 of these labs.

The mean of the results of the remaining 38 labs is $6.1 \pm 1.0 \text{ mg/kg}$. The relative standard deviation is 17 percent.

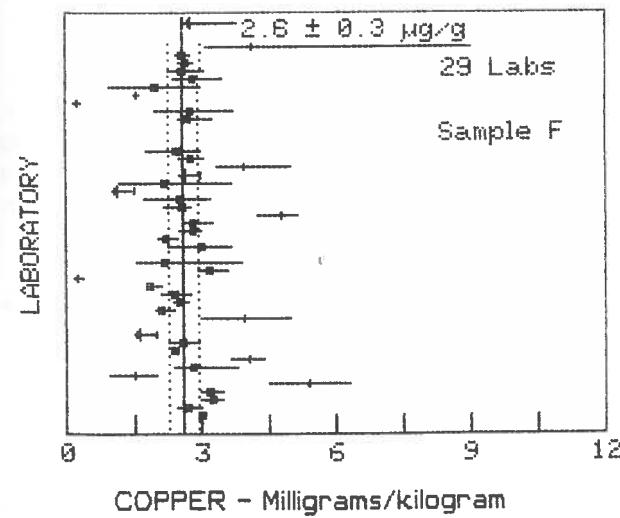


42 labs submitted results ranging from less than 0.1 to 7.5 mg/kg.

35 labs submitted quantitative values with a range of 0.19 to 7.5 and a mean of $1.7 \pm 1.2 \text{ mg/kg}$.

A *t* test rejected the results of 8 of these labs.

The mean of the results of the remaining 27 labs is $1.2 \pm 0.2 \text{ mg/kg}$. The relative standard deviation is 20 percent.



42 labs submitted results ranging from 0.18 to 9.0 mg/kg.

39 labs submitted quantitative values with a range of 0.18 to 9.0 and a mean of $2.7 \pm 1.0 \text{ mg/kg}$.

A *t* test rejected the results of 10 of these labs.

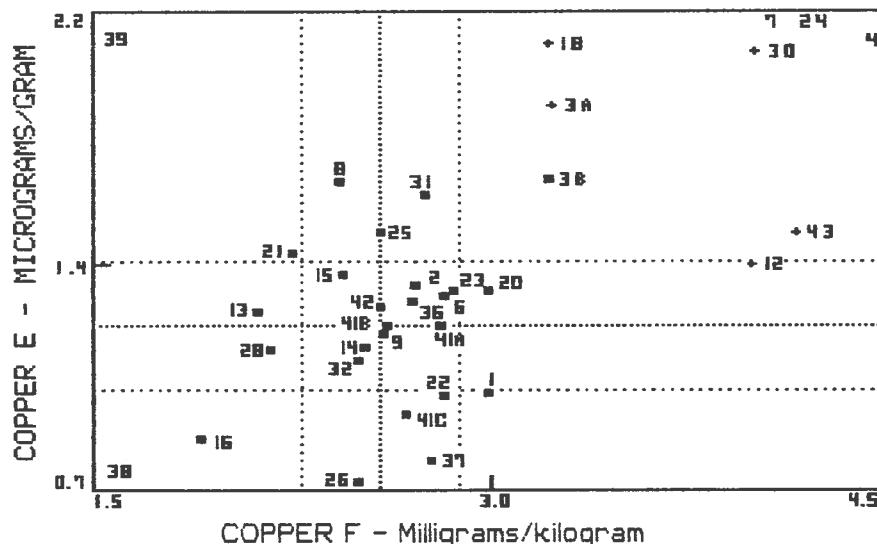
The mean of the results of the remaining 29 labs is $2.6 \pm 0.3 \text{ mg/kg}$. The relative standard deviation is 13 percent.

Three-quarters of the labs (31) employed an open beaker digestion using nitric acid or aqua regia (8), or a combination of nitric acid and another oxidizing acid such as sulphuric (3) or perchloric (20) acid. Almost all others used some form of dry ashing procedure. Two labs used a closed vessel acid decomposition (BOMB). This is in marked contrast to the ICES, largely European, experience (Berman, 1984) where one-third of the 49 participants employed some form of closed vessel decomposition.

Slightly more than half the labs (22) used flame atomic absorption spectrometry (FAAS) and about another twenty-five percent (10) used inductively coupled plasma atomic emission spectrometry (ICP) in their measurement procedures (in contrast to the ICES study where only one European lab employed this technique). Seventeen percent utilized graphite furnace atomic absorption spectrometry (GFAAS), and one each used electroanalytical techniques (ASV), direct current plasma atomic emission spectrometry (DCP) and isotope dilution mass spectrometry (IDMS).

There is no apparent correlation between performance and methodology as there are insufficient data for any particular procedure to demonstrate that it is better or worse than any of the others used by the participants. Seven of the eight rejected results for Sample E are higher than the mean. Six of the eight and ten sets of means rejected for Samples E and F respectively are from the same labs. Oddly enough, two of the three labs whose results are rejected for Sample D do quite well with the other two samples with lower concentrations.

COPPER E vs COPPER F



Zinc

Only three laboratories did not submit results for zinc. The data for zinc are the best of all the six metals studied. This is probably a reflection of the relatively high concentrations of zinc in the samples compared to those of the other five metals. Summaries of the results are listed beside the graphs on page 8 and the overall final means and standard deviations are shown in Table II below.

Table II

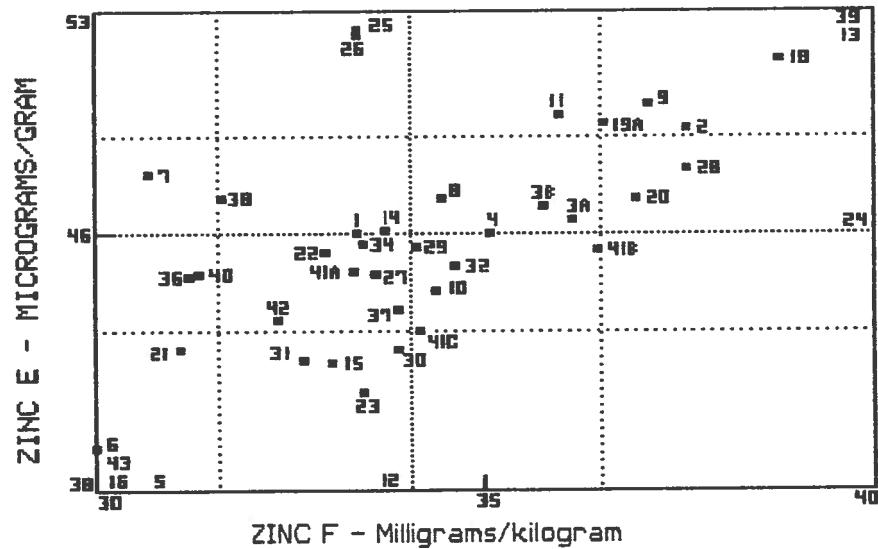
Zinc Concentrations - mg/kg

Sample D	27.9	\pm 2.3	(8%)	43, 5
Sample E	46.0	\pm 3.0	(7%)	43, 6
Sample F	34.0	\pm 2.4	(7%)	43, 3

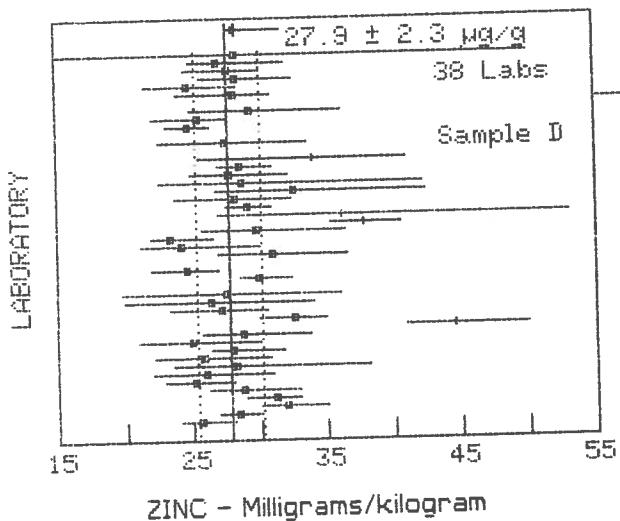
Sample preparation methods are identical to those for copper.

Metal concentration measurement procedures are also largely similar to those for copper, except that in probable recognition of the higher zinc concentrations and the good sensitivity of atomic absorption spectrometry for this metal all but two of the AAS measurements were by flame atomic absorption spectrometry. Two labs also used instrumental neutron activation analysis (INAA) which requires no sample decomposition.

ZINC E vs ZINC F



ZINC DATA

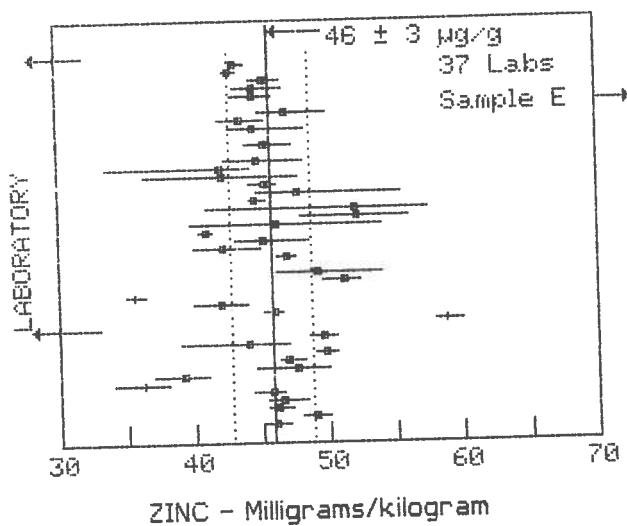


43 labs submitted results ranging from 15 to 68.4 mg/kg.

All labs submitted quantitative values with a mean 29.8 ± 7.2 mg/kg.

A *t* test rejected the results of 5 of these labs.

The mean of the results of the remaining 38 labs 27.9 ± 2.3 mg/kg. The relative standard deviation 8 percent.

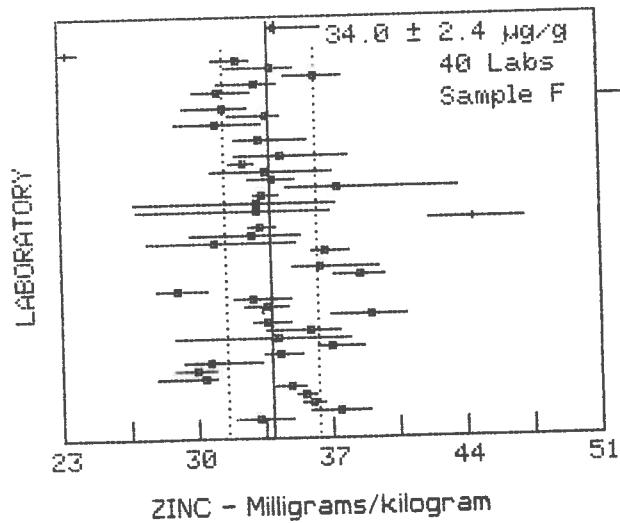


43 labs submitted results ranging from 25 to 106 mg/kg.

All labs submitted quantitative values with a mean 46.0 ± 11.0 mg/kg.

A *t* test rejected the results of 6 of these labs.

The mean of the results of the remaining 37 labs 46.0 ± 3.0 mg/kg. The relative standard deviation 7 percent.



43 labs submitted results ranging from 23 to 98.8 mg/kg.

All labs submitted quantitative values with a mean 35.5 ± 10.4 mg/kg.

A *t* test rejected the results of 3 of these labs.

The mean of the results of the remaining 40 labs 34.0 ± 2.4 mg/kg. The relative standard deviation 7 percent.

The plot of Sample E against Sample F on page 7 shows no sets of erratic results. The general distribution is more normal for this type of graph than that encountered with copper.

The submitted zinc data are listed on pages 29 to 33 along with abbreviated methodologies.

Arsenic

Only twenty-five (less than sixty percent of the participants) submitted results for arsenic. This is probably indicative of the fact that fewer laboratories routinely analyze samples for this metal. Summaries of the results for this element are listed beside the graphs on page 10 and the overall final means and standard deviations are shown in Table III below. The two numbers in the final column of each table represent the number of quantitative values submitted and values excluded respectively.

TABLE III

Arsenic Concentrations - mg/kg

Sample D	30.8 ± 4.2 (14%)	24, 5
Sample E	5.9 ± 0.9 (16%)	22, 5
Sample F	4.0 ± 1.0 (24%)	21, 3

The almost universal method of sample decomposition was to use nitric acid along with some other oxidizing acid such as sulphuric or perchloric, or a mixture of all three (although one lab used only nitric acid and another only hydrochloric acid). Two labs employed dry ashing procedures and one lab used a bomb technique.

Half of the laboratories utilized a hydride generation method followed by an atomic absorption spectrometric measurement (HG-AAS) in order to assay the arsenic contents. Three labs did direct measurements by GFAAS (one with Ni as a matrix modifier), three used INAA, and another three generated the hydride and determined the arsenic content using a silver diethyldithiocarbamate spectrophotometric procedure (HG-AgDDC). Four labs atomized the arsenic solutions directly into a plasma for spectrometric measurement and a fifth generated the hydride which was then introduced into the plasma.

Fourteen to twenty-three percent of the results were rejected in order to achieve the overall means shown above. Even then, the distribution for sample F is very large, a relative standard deviation of 24 percent representing a range of almost a factor of three from lowest to highest accepted values.

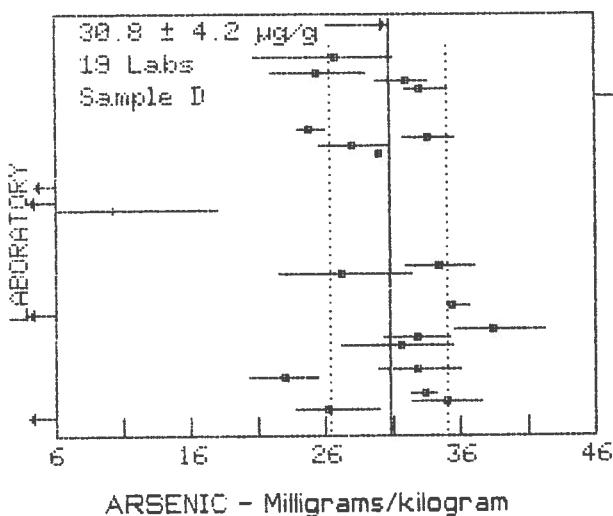
Two of the three labs which employed the AgDDC procedure produced unacceptable values. However, one of these labs used only a hydrochloric acid leach to decompose the samples which is insufficient to release all the arsenic. All other rejected results were from hydride generation AAS methods, one-third of all values submitted using this technique.

There is no apparent correlation between sample preparation procedures (except for the hydrochloric acid leach) and performance. All rejected results came from five laboratories, three of which produced poor results for all samples.

The plot of Sample E against Sample F on page 11 shows no indication of erratic performance.

The submitted arsenic data are listed on pages 33 to 36 along with abbreviated methodologies.

ARSENIC DATA

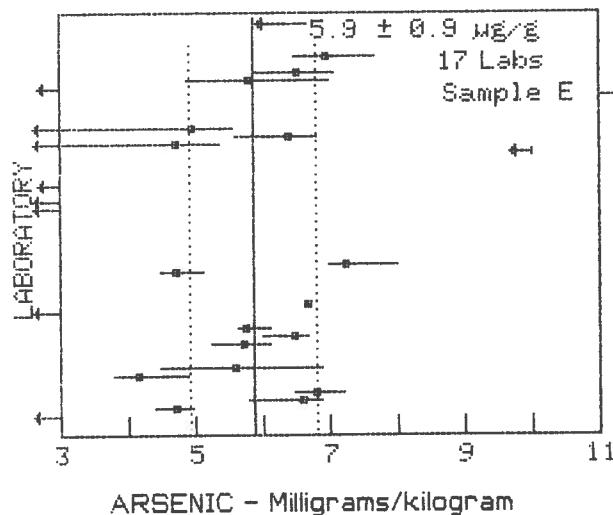


25 labs submitted results ranging from less than 0 to 113 mg/kg.

24 labs submitted quantitative values with a range of 0.2 to 113 and a mean of 29.8 ± 20.9 mg/kg.

A *t* test rejected the results of 5 of these labs.

The mean of the results of the remaining 19 labs is 30.8 ± 4.2 mg/kg. The relative standard deviation is 14 percent.

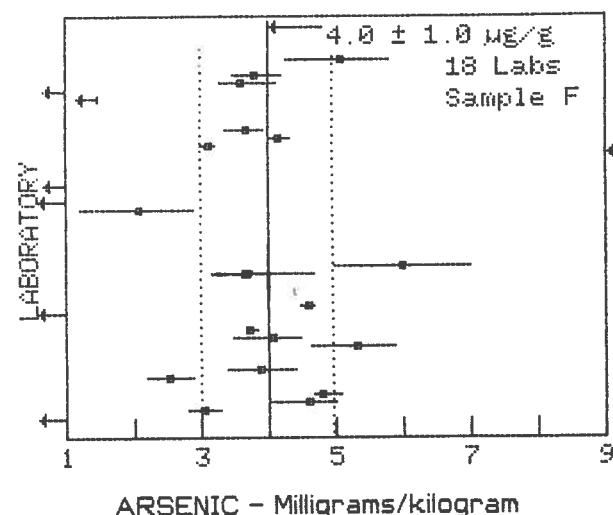


25 labs submitted results ranging from 0.042 to 36 mg/kg.

22 labs submitted quantitative values with a range of 0.042 to 36.9 and a mean of 6.3 ± 7.2 mg/kg.

A *t* test rejected the results of 5 of these labs.

The mean of the results of the remaining 17 labs is 5.9 ± 0.9 mg/kg. The relative standard deviation is 16 percent.



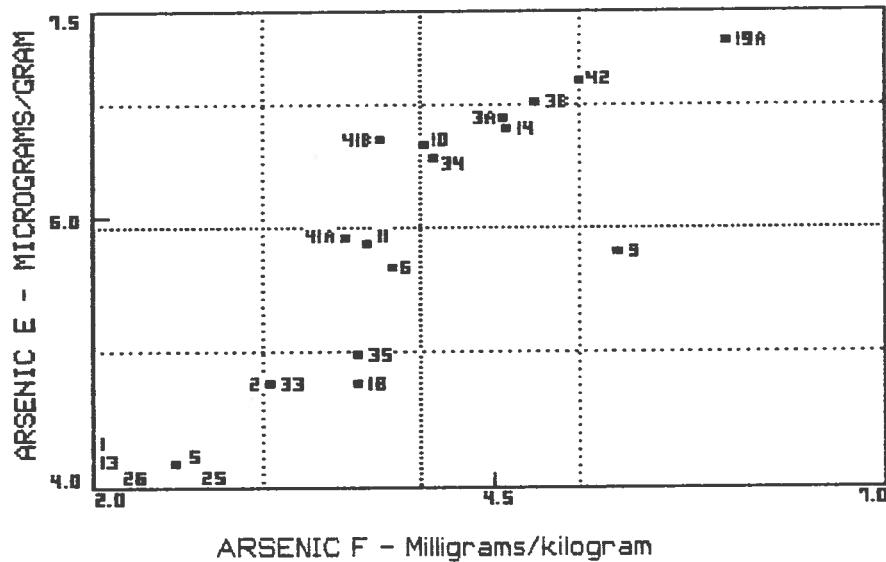
25 labs submitted results ranging from less than 0 to less than 10 mg/kg.

21 labs submitted quantitative values with a range of 0.106 to 7 and a mean of 3.5 ± 1.6 mg/kg.

A *t* test rejected the results of 3 of these labs.

The mean of the results of the remaining 18 labs is 4.0 ± 1.0 mg/kg. The relative standard deviation is 24 percent.

ARSENIC E vs ARSENIC F



Cadmium

Although only 5 laboratories did not submit results for cadmium, there were only 19, 36 and 34 quantitative values for the three samples respectively. This is due to the inherently low concentrations of cadmium in these materials and apparent lack of sensitivity in many of the laboratories' procedures.

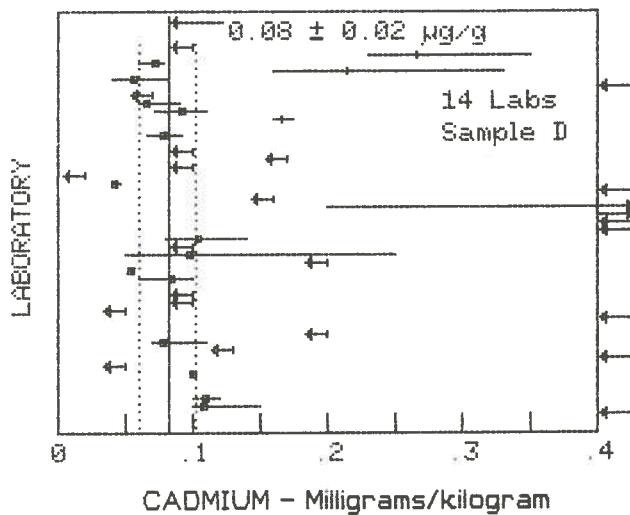
Summaries of the results for this element are listed beside the graphs on page 12 and the overall final means and standard deviations are shown in Table IV below.

TABLE IV
Cadmium Concentrations - mg/kg

Sample D	0.08 ± 0.02 (26%)	19, 5
Sample E	0.48 ± 0.06 (13%)	36, 7
Sample F	0.31 ± 0.06 (19%)	34, 4

It is apparent, in spite of the low concentrations, that almost three-quarters of the participants can produce valid results at the 0.3 to 0.4 mg/kg level and that about one-third of them do well even at the 0.08 mg/kg level.

CADMIUM DATA

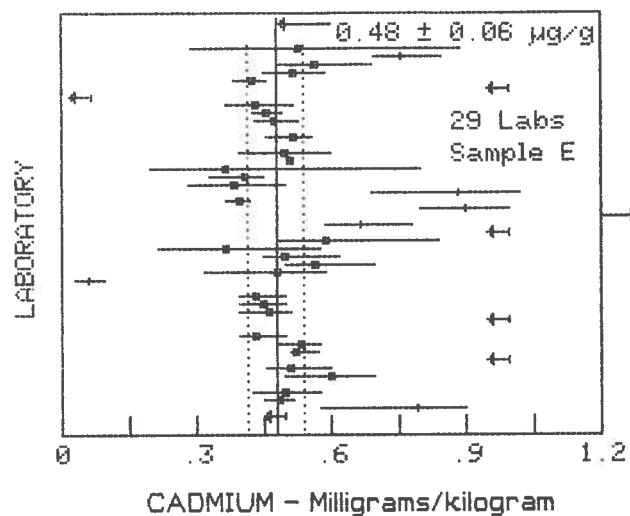


41 labs submitted results ranging from less than 0.04 to 4.4 mg/kg.

19 labs submitted quantitative values with a range of 0.04 to 4.4 and a mean of 0.32 ± 0.84 mg/kg.

A *t* test rejected the results of 5 of these labs.

The mean of the results of the remaining 14 labs is 0.08 ± 0.02 mg/kg. The relative standard deviation is 26 percent.

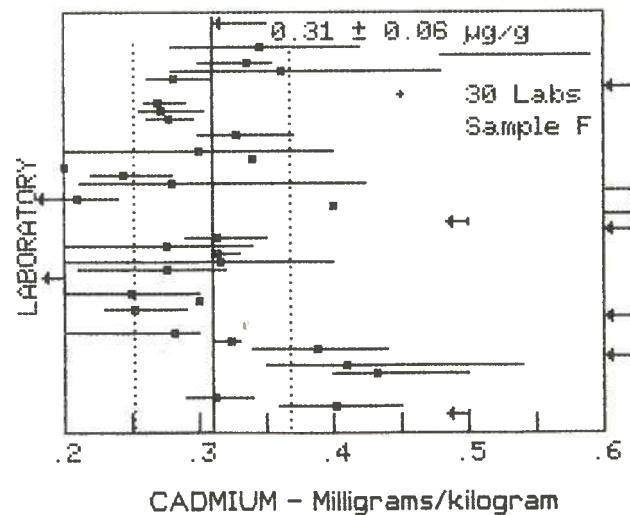


42 labs submitted results ranging from 0.03 to 5.4 mg/kg.

36 labs submitted quantitative values with a range of 0.03 to 5.4 and a mean of 0.64 ± 0.73 mg/kg.

A *t* test rejected the results of 7 of these labs.

The mean of the results of the remaining 29 labs is 0.48 ± 0.06 mg/kg. The relative standard deviation is 13 percent.



41 labs submitted results ranging from less than 0.18 to 4.2 mg/kg.

34 labs submitted quantitative values with a range of 0.18 to 4.2 and a mean of 0.44 ± 0.63 mg/kg.

A *t* test rejected the results of 4 of these labs.

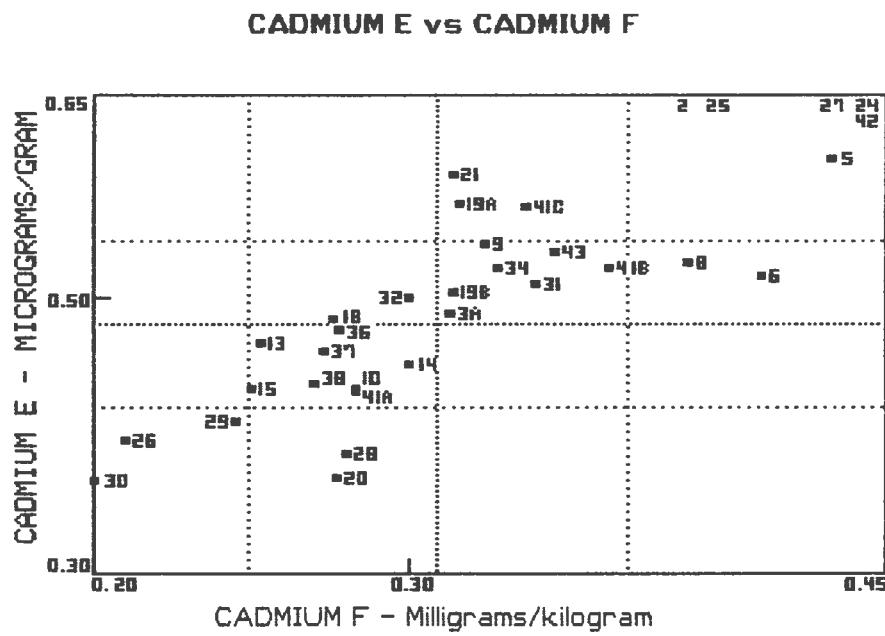
The mean of the results of the remaining 30 labs is 0.31 ± 0.06 mg/kg. The relative standard deviation is 19 percent.

Sample preparation procedures for cadmium were essentially similar to those described for copper.

About 40 percent (16) of the participants used flame atomic absorption spectrometry (FAAS) and another one-third (14) graphite furnace atomic absorption spectrometry (GFAAS) to measure the metal concentrations. Nine introduced the metal solutions into a plasma (ICP or DCP), two employed electroanalytical procedures (ASV) and one used isotope dilution mass spectrometry.

However, only three of the sixteen FAAS labs were able to produce a quantitative result for Sample D, and one of these was rejected. There were only two quantitative values from the plasma excitation labs for the same sample and both of these were rejected. This indicates that GFAAS was the sole common and dependable measurement system capable of analysis of these materials for cadmium at levels less than 0.1 mg/kg. The distribution of accepted results for Sample D is relatively large (RSD equals 26 percent) but this must be taken within the context of the low cadmium concentration and is comparable to results achieved in the ICES study for similar concentrations in biological tissues.

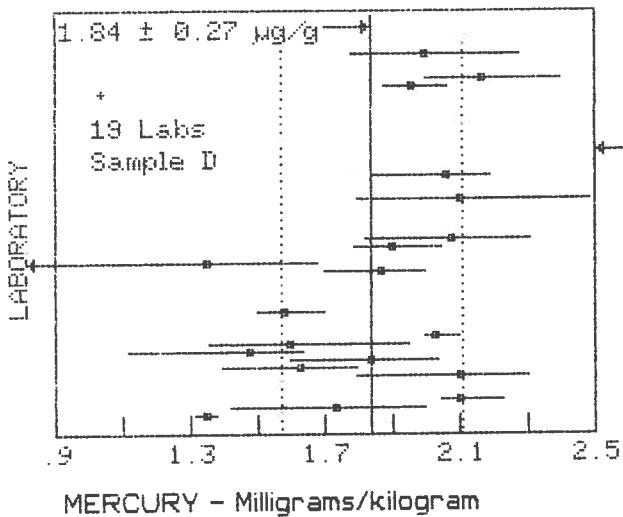
The situation changes dramatically for concentrations at and above the 0.3 mg/kg level. For these concentrations there were a dozen FAAS and seven plasma sets of quantitative values for each of Samples D and E with only a few poor values among them. Two of the FAAS procedures separate and concentrate the cadmium prior to measurement but neither of these labs submitted quantitative information regarding Sample D.



The above plot of Sample E against Sample F shows no sets of erratic results. However, laboratories with problems tend towards a high bias. There is no apparent correlation between methodologies and performance for Samples E and F.

The submitted cadmium data are listed on pages 36 to 39 along with abbreviated methodologies.

MERCURY DATA

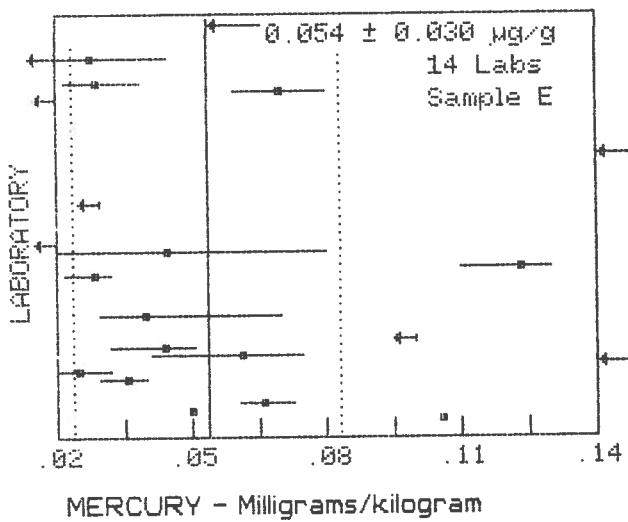


21 labs submitted results ranging from 0.88 to less than 4 mg/kg.

20 labs submitted quantitative values with a range of 0.88 to 2.49 and a mean of $1.80 \pm 0.32 \text{ mg/kg}$.

A *t* test rejected the results of 1 of these labs.

The mean of the results of the remaining 19 labs is $1.84 \pm 0.27 \text{ mg/kg}$. The relative standard deviation is 15 percent.



Mercury

Only one-half of the responding laboratories submitted results for mercury.

Summaries of the results for this element are listed beside the graphs on page 14 and the overall final means and standard deviations are shown in Table V below. The results for Sample E are bracketted in order to emphasize that the mean of 0.054 mg/kg cannot be considered to be a reliable estimate of the concentration of mercury in this sample due to the inordinately large distribution of values from which it is calculated.

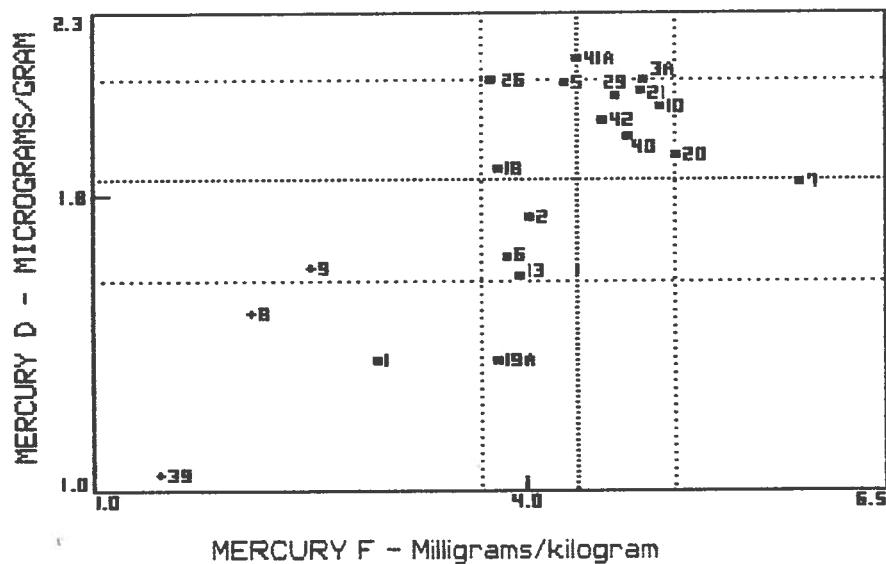
TABLE V
Mercury Concentrations - mg/kg

Sample D	1.84 ± 0.27	(15%)	20, 1
(Sample E)	0.054 ± 0.030	(56%)	14, 0
Sample F	4.38 ± 0.67	(15%)	20, 3

All participants decomposed the samples using an oxidizing acid medium. These ranged from simple aqua regia to mixtures of nitric with sulphuric and/or perchloric acid and other oxidants such as persulphate, dichromate or permanganate.

The universal method of measurement (except for one lab) was cold vapour atomic absorption spectrometry (CVAAS) following reduction of the mercury in solution, usually by a tin (II) compound or in a few cases by sodium borohydride. One laboratory used inductively coupled plasma atomic emission (ICP) but did not report quantitative values.

MERCURY D vs MERCURY F



The results for Samples D and F are good, indicating that the great majority of the labs that analyze for mercury do well at levels above 1 mg/kg. Performance for Sample E is disappointing, especially in view of the relatively good performance at this concentration level in the ICES study. The distribution of results from the fourteen labs is such that none of the values were rejected by the *t* test. This is an example of a situation where there is just insufficient data to draw reliable conclusions. Intuitively, we feel that the concentration of mercury in Sample E is between 0.03 and 0.04 mg/kg but this cannot be substantiated by the data at hand.

The mercury concentrations in Samples E and F are too disparate to warrant their plotting in the Youden manner. For this reason the plot of Sample D against Sample F is displayed on page 15. Only one lab seems to be consistently low for both samples.

The submitted mercury data are listed on pages 39 to 42 along with abbreviated methodologies.

Lead

Although 40 laboratories submitted quantitative results for lead in Sample D, there were only 20 and 23 quantitative sets of values for the other two samples respectively. This is due to the inherently low concentrations of lead in the latter materials and an apparent lack of sensitivity in many of the laboratories' procedures.

Summaries of the results for this element are listed beside the graphs on page 17 and the overall final means and standard deviations are shown in Table VI below. The results for Sample E are bracketted in order to emphasize that the mean of 0.21 mg/kg cannot be considered to be a reliable estimate of the concentration of mercury in this sample due to the inordinately large distribution of values from which it is calculated.

TABLE VI
Lead Concentrations - mg/kg

Sample D	9.0 ± 2.1 (24%)	40, 5
(Sample E)	0.21 ± 0.12 (56%)	20, 9)
Sample F	0.24 ± 0.06 (26%)	23,10

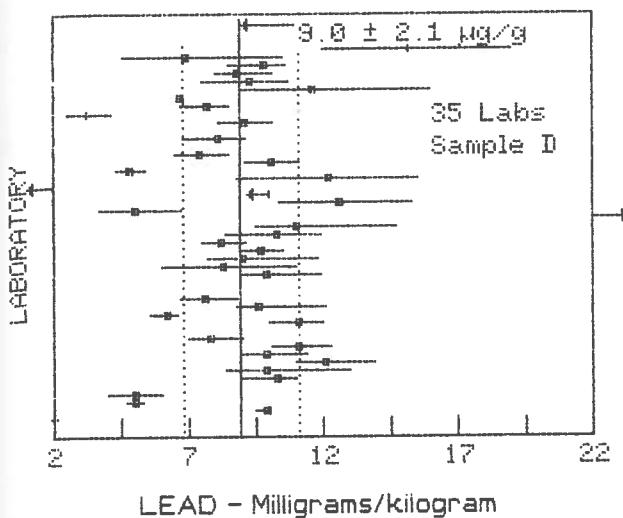
Sample decomposition procedures are essentially identical to those used for copper.

More than one-third (15) of the participants used flame atomic absorption spectrometry (FAAS) and about another third (13) graphite furnace atomic absorption spectrometry (GFAAS) in order to measure the lead concentrations in solution. Nine labs used plasma techniques (8 ICP and 1 DCP), two employed electroanalytical methods and one isotope dilution mass spectrometry (IDMS). Three of the labs that employed FAAS concentrated the lead by chelation/solvent extraction prior to determination.

Results for Sample D are fair, indicating that the majority of the participants can analyze for lead in fish muscle at the 10 mg/kg level.

Seven labs utilizing FAAS without prior preconcentration of the lead submitted quantitative values for Samples E and F. All of these were rejected by the *t* test. One of the three that preconcentrated the lead was rejected for Sample F.

LEAD DATA

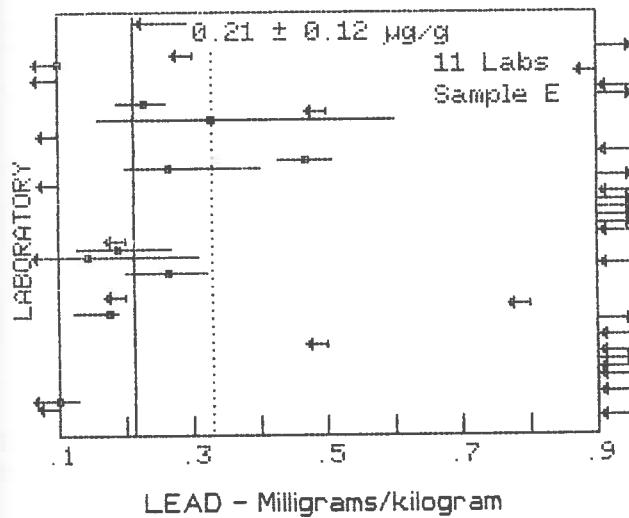


41 labs submitted results ranging from 0.33 to 73.5 mg/kg.

40 labs submitted quantitative values with a range of 0.33 to 73.5 and a mean of $10.0 \pm 9.6 \text{ mg/kg}$.

A *t* test rejected the results of 5 of these labs.

The mean of the results of the remaining 35 labs is $9.0 \pm 2.1 \text{ mg/kg}$. The relative standard deviation is 24 percent.

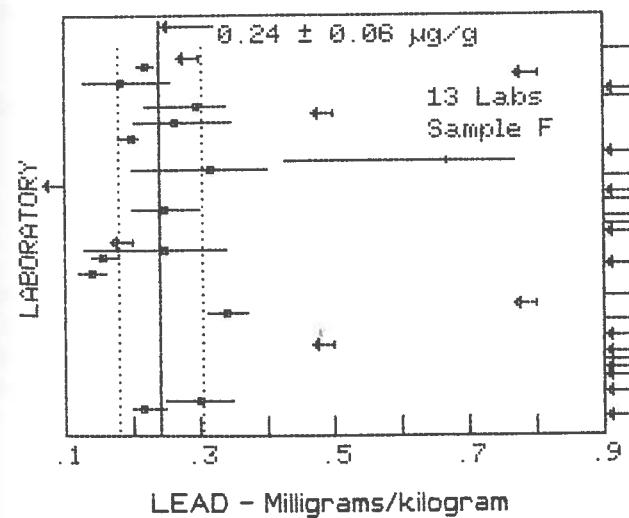


41 labs submitted results ranging from less than 0.05 to 64.6 mg/kg.

20 labs submitted quantitative values with a range of 0.05 to 64.6 and a mean of $4.43 \pm 13.43 \text{ mg/kg}$.

A *t* test rejected the results of 9 of these labs.

The mean of the results of the remaining 11 labs is $0.21 \pm 0.12 \text{ mg/kg}$. The relative standard deviation is 56 percent.



41 labs submitted results ranging from less than 0.05 to 60.5 mg/kg.

23 labs submitted quantitative values with a range of 0.12 to 60.5 and a mean of $3.52 \pm 11.33 \text{ mg/kg}$.

A *t* test rejected the results of 10 of these labs.

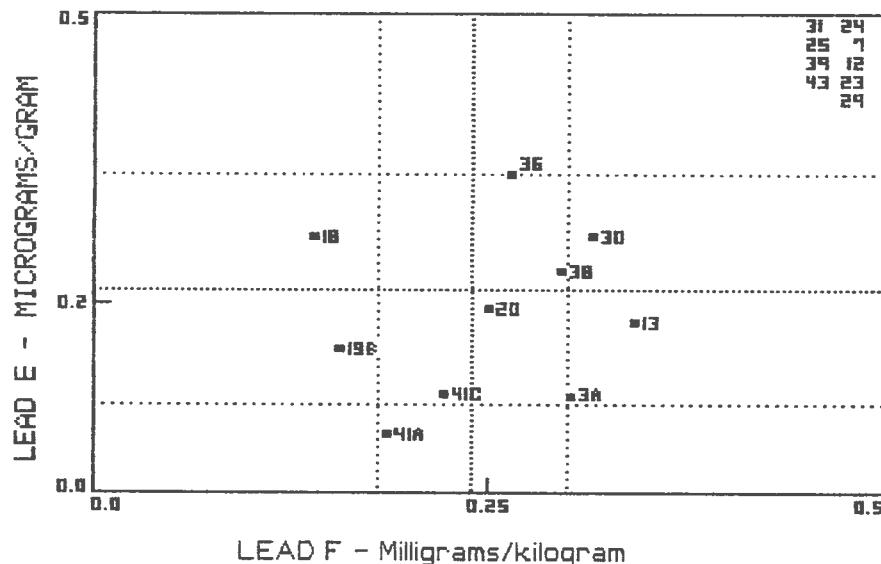
The mean of the results of the remaining 13 labs is $0.24 \pm 0.06 \text{ mg/kg}$. The relative standard deviation is 26 percent.

There were no quantitative ICP results for Sample E and only one for Sample F, which was rejected. There was one DCP result for each of these samples. The value submitted for Sample E was rejected.

This leaves six and eight GFAAS values for the two samples respectively, of which none were rejected. One of the two ASV results survived for each sample as well as the IDMS results.

The obvious conclusion is that FAAS, ICP and DCP methods are, in general, not sensitive enough to determine lead in biological tissues when the concentration is less than 1 mg/kg unless there is prior preconcentration. This is in line with earlier findings with respect to FAAS in ICES studies (Topping, 1982, Harms et. al., 1982).

LEAD E vs LEAD F



Why is the distribution of results for Sample E so much worse than that for Sample F even though the concentrations of lead in both are apparently very close? There is no evidence from the submitted data that Sample E is less homogeneous with respect to lead than Sample F. We believe that the true value for the concentration of lead in this sample is probably closer to 0.1 mg/kg (as indicated by the stable isotope dilution mass spectrometric results), a level at which the sensitivity and blank problems in many labs may outweigh their ability to provide reliable results. More data are needed substantiate this argument.

The submitted data for lead are listed on pages 42 to 45 along with abbreviated methodologies.

Other Metals

Results for 25 other metals were received. For nineteen of these there were only one or two results. The means and standard deviations were calculated for the remaining six elements (sodium, calcium, chromium, manganese, iron and nickel). The *t* test was not applied to these data. These are listed on the following pages in Tables VII to XII. The submitted data are on pages 46 to 48. Note that the sodium concentrations are in percent.

TABLE VII
Sodium Concentrations - percent

$n = 3$

Sample D	0.303 ± 0.012 (4%)
Sample E	1.09 ± 0.10 (9%)
Sample F	0.304 ± 0.014 (5%)

TABLE VIII
Calcium Concentrations - mg/kg

$n = 3$

Sample D	255 ± 7 (3%)
Sample E	562 ± 35 (6%)
Sample F	231 ± 46 (20%)

TABLE IX
Chromium Concentrations - mg/kg

$n = 6$

Sample D	0.86 ± 0.28 (32%,
Sample E	1.02 ± 0.13 (13%)
Sample F	1.36 ± 0.35 (26%)

The chromium results for Sample D from lab 34 were arbitrarily removed from the above calculations. There are only 5 sets of results for this sample.

TABLE X
Manganese Concentrations - mg/kg

$n = 4$

Sample D	3.94	\pm	0.95	(24%)
Sample E	0.88	\pm	0.20	(23%)
Sample F	0.51	\pm	0.09	(19%)

TABLE XI
Iron Concentrations - mg/kg

$n = 6$

Sample D	125	\pm	9	(7%)
Sample E	16.2	\pm	1.0	(6%)
Sample F	29.2	\pm	2.3	(8%)

TABLE XII
Nickel Concentrations - mg/kg

$n = 3$

Sample D	2.37	\pm	1.16	(49%)
Sample E	1.23	\pm	0.59	(48%)
Sample F	1.10	\pm	0.33	(30%)

There are too few results for any of the above metals to warrant serious general conclusions. The sodium and iron results for all samples and the calcium concentrations for Samples D and E are probably valid concensus values.

SUMMARIES

The results for the six core metals in the three samples are summarized in Table XIII on page 24.

Table XIV on page 25 is an attempt to show the pattern of results achieved by each laboratory for the six metals in the three samples.

The list of participating laboratories is on pages 22 and 23.

CONCLUSIONS

This exercise has demonstrated that the good majority of the participants can produce comparable and accurate data for the trace metals copper and zinc at levels found in marine biological tissues. There are also generally good capabilities for cadmium and fair for lead where the respective metal concentrations are greater than a few tenths and several milligrams per kilogram of tissue.

Less than half of the labs (19) produced quantitative results for cadmium at the 0.08 mg/kg level. Three quarters of these were quite satisfactory. Flame atomic absorption and plasma emission spectrometric techniques are generally not sensitive enough for this concentration for cadmium.

Also, only about half the labs (20-23) submitted quantitative values for lead at the less than 0.3 mg/kg level. Of these, only about one half (13) produced valid results for lead in Sample F and, we feel, about six labs for lead in Sample E whose concentration may be around the 0.1 mg/kg level. As in the case of cadmium, the flame atomic absorption and plasma emission methods are not sensitive enough to analyze for lead at these concentrations without prior concentration.

Less than sixty percent of the participants generally analyze for arsenic. However, the majority of these can apparently analyze for this element at the concentrations present in marine biological tissues.

Less than fifty percent of the labs submitting results analyzed the samples for mercury. Performance was very good at levels greater than one mg/kg of mercury but only about a third of these were able to cope with the low level mercury concentration of less than 0.05 mg/kg in the scallops sample (E).

The mercury and lead means calculated for Sample E should not be considered as reliable estimates of the concentrations of these metals in the sample. Other consensus means are probably fairly reliable concentration estimates.

Sample preparation techniques used by the participants, with but few exceptions, appear to be adequate for the six metals in marine biological tissues. It is possible that there are contamination problems in some laboratories that obviate good lead and cadmium analyses at the levels of occurrence in the scallops sample.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the help of Dr Robin Law of the Fisheries Laboratory, Burnham-on-Crouch, in supplying the dogfish sample (D) and of Dr. Graham Bligh, Technical University of Nova Scotia, in preparing Samples E and F. Margaret Miedema and Vincent Clancy helped with bottling and packaging.

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TABLE XIII
SUMMARY OF RESULTS

milligrams per kilogram

SAMPLE D

	<u>Copper</u>	<u>Zinc</u>	<u>Arsenic</u>	<u>Cadmium</u>	<u>Mercury</u>	<u>Lead</u>
Total range	0.3-16.4	15-68.4	<0.1-113	<0.02-4.4	0.88-<4	0.33-73.5
No. of labs	42	43	25	41	21	41
Quant. values	0.3-16.4	15-68.4	0.2-113	0.04-4.4	0.88-2.49	0.33-73.5
No. of labs	41	43	24	19	20	40
Mean	6.0 ± 1.6	29.8 ± 7.2	29.8 ± 20.9	0.32 ± 0.84	1.80 ± 0.32	10.0 ± 9.6
After <i>t</i> test	3.0-11.2	15-50	20.5-42.3	0.040-0.25	0.88-2.49	3.7-16
No. of labs	38	38	19	14	19	35
Mean	6.1 ± 1.0	27.9 ± 2.3	30.8 ± 4.2	0.08 ± 0.02	1.84 ± 0.27	9.0 ± 2.1
RSD	17%	8%	14%	26%	15%	24%

SAMPLE E

	<u>Copper</u>	<u>Zinc</u>	<u>Arsenic</u>	<u>Cadmium</u>	<u>Mercury</u>	<u>Lead</u>
Total range	<0.1-7.5	25-106	0.042-36.9	0.03-5.4	<0.01-<4	<0.05-64.6
No. of labs	42	43	25	42	20	41
Quant. values	0.19-7.5	25-106	0.042-36.9	0.03-5.4	0.018-0.13	0.05-64.6
No. of labs	35	43	22	36	14	20
Mean	1.7 ± 1.2	46.0 ± 11.0	6.3 ± 7.2	0.64 ± 0.73	0.054 ± 0.030	4.43 ± 13.43
After <i>t</i> test	0.276-2	33.7-57.5	2.57-8	0.2-0.84	0.018-0.08	0.05-0.603
No. of labs	27	37	17	29	14	11
Mean	1.2 ± 0.2	46.0 ± 3.0	5.9 ± 0.9	0.48 ± 0.06	(0.054 ± 0.030);	(0.21 ± 0.12)
RSD	20%	7%	16%	13%	56%	56%

SAMPLE F

	<u>Copper</u>	<u>Zinc</u>	<u>Arsenic</u>	<u>Cadmium</u>	<u>Mercury</u>	<u>Lead</u>
Total range	0.18-9	23-98.8	<0.1-<10	<0.02-4.2	1.47-6.59	<0.05-60.5
No. of labs	42	43	25	41	21	41
Quant. values	0.18-9	23-98.9	0.106-7	0.18-4.2	1.47-6.59	0.12-60.5
No. of labs	39	43	21	34	20	23
Mean	2.7 ± 1.0	35.5 ± 10.4	3.5 ± 1.6	0.44 ± 0.63	4.03 ± 1.07	3.52 ± 11.33
After <i>t</i> test	1-3.68	26.9-43.8	1.2-7	0.18-0.54	3.0-6.59	0.12-0.4
No. of labs	29	40	18	30	17	13
Mean	2.6 ± 0.3	34.0 ± 2.4	4.0 ± 1.0	0.31 ± 0.06	4.38 ± 0.67	0.24 ± 0.06
RSD	13%	7%	24%	19%	15%	26%

TABLE XIV

PATTERN OF RESULTS

<u>Lab</u>	<u>Copper</u>	<u>Zinc</u>	<u>Arsenic</u>	<u>Cadmium</u>	<u>Mercury</u>	<u>Lead</u>
1	** ** **	** ** **	RT RT RT	LD LD LD	** ** **	RT LD LD
2	** ** **	** ** **	** ** **	** RT **	** ** **	** LD **
3A	** RT **	** ** **	** ** **	** ** **	** ** **	** ** **
3B	** ** **	** ** **	** ** **	-- ** --	-- -- --	** LD LD
4	** RT RT	** ** **	-- -- --	-- -- --	-- -- --	-- -- --
5	** LD RT	** RT **	** ** **	** ** **	** ** **	** LD LD
6	** ** **	** ** **	** ** **	LD ** **	** ** **	** LD LD
7	** RT RT	** ** **	-- -- --	LD LD LD	** LD **	** RT RT
8	** ** **	** ** **	-- -- --	LD ** **	** ** RT	** LD LD
9	** ** **	** ** **	** ** **	** ** **	** ** RT	** LD LD
10	** LD LD	** ** **	** ** **	LD ** **	** LD **	** LD LD
11	-- -- --	** ** **	** ** **	-- -- --	-- -- --	-- -- --
12	** ** RT	RT RT **	-- -- --	LD LD LD	-- -- --	** RT RT
13	** ** **	** RT **	RT RT RT	LD ** **	** ** **	** ** **
14	** ** **	** ** **	** ** **	LD ** **	-- -- --	** LD LD
15	** ** **	** ** **	-- -- --	LD ** **	-- -- --	** LD RT
16	** ** **	** RT **	-- -- --	-- -- --	-- -- --	-- -- --
17	RT LD RT	-- -- --	-- -- --	** RT LD	-- -- --	-- -- --
18	** RT **	** ** **	** ** **	** ** **	** ** **	** ** **
19A	** LD **	** ** **	** ** **	LD ** **	** ** **	** LD LD
19B	-- -- --	-- -- --	-- -- --	** ** **	-- -- --	** ** **
20	** ** **	** ** **	-- -- --	LD ** **	** ** **	** ** **
21	** ** **	** ** **	-- -- --	** ** **	** LD **	** LD LD
22	** ** **	** ** **	-- -- --	LD LD LD	-- -- --	** LD LD
23	** ** **	** ** **	-- -- --	LD RT LD	-- -- --	** RT RT
24	** RT RT	RT ** RT	-- -- --	RT RT RT	-- -- --	RT RT RT
25	RT ** **	RT ** **	RT RT **	RT RT **	-- -- --	** RT **
26	** ** **	** ** **	RT RT RT	LD ** **	** LD **	** RT RT
27	** LD LD	** ** **	-- -- --	LD RT RT	-- -- --	LD LD LD
28	RT ** **	** ** **	LD LD LD	** ** **	-- -- --	RT LD LD
29	LD LD LD	** ** **	-- -- --	LD ** **	** -- **	** RT RT
30	** RT RT	** ** **	-- -- --	LD ** **	-- -- --	** ** **
31	** ** **	** ** **	-- -- --	LD ** **	-- -- --	** ** RT
32	** ** **	RT ** **	** LD LD	LD ** **	LD LD LD	** LD LD
33	-- -- --	-- -- --	** ** **	-- -- --	-- -- --	-- -- --
34	-- -- --	** ** **	** ** **	** ** **	-- -- --	** LD **
35	-- -- --	-- -- --	** ** **	-- -- --	-- -- --	-- -- --
36	** ** **	** ** **	-- -- --	RT ** **	-- -- --	** ** **
37	** ** **	** ** **	-- -- --	** ** **	-- -- --	RT LD LD
38	** RT RT	** ** **	-- -- --	** ** **	-- -- --	** ** **
39	** RT RT	RT RT RT	RT RT LD	LD LD RT	RT LD RT	** RT RT
40	** LD **	** ** **	** LD LD	LD LD LD	** ** **	** LD LD
41A	** ** **	** ** **	** ** **	** ** **	** ** **	** ** **
41B	** ** **	** ** **	** ** **	RT ** **	-- -- --	** LD LD
41C	** ** **	** ** **	-- -- --	** ** **	-- -- --	** ** **
42	** ** **	** ** **	** ** **	RT RT RT	** ** **	** LD LD
43	** ** RT	** RT RT	-- -- --	LD ** **	-- -- --	RT RT RT

** Results reported and accepted by a *t* test.

LD Results reported but less than a limit of determination.

RT Results reported but rejected by a *t* test.

-- No results reported.

COPPER DATA

Lac	Mean	RSD							
1			HCl-HNO ₃ -HClO ₄ /HF-HCl-FAAS						
	7.3	15.7	8	8	6				
	1.0	0.0	1	1	1				
	3.0	0.0	3	3	3				
2			HNO ₃ -HClO ₄ -FAAS						
	6.5	13.1	6.3	6.2	5.1	6.5	7.2	7.5	
	1.3	15.5	1.5	1.3	1.0	1.2	1.5	1.5	
	2.8	8.2	2.5	2.6	2.9	3.0	2.9	2.5	
3A			BOMB/HNO ₃ /HClO ₄ -GFAAS						
	6.8	10.3	6.9	7.0	7.9	6.0	6.1	6.6	
	1.9 ^{RT}	10.0	1.9	1.9	2.1	1.8	2.1	1.6	
	3.3	6.7	3.4	3.2	3.0	3.5	3.0	3.4	
3B			BOMB/HNO ₃ /HClO ₄ -ICP						
	7.0	10.6	7.7	7.6	6.6	5.9	7.5	6.5	
	1.7	9.8	1.5	1.9	1.5	1.7	1.6	1.8	
	3.2	5.8	3.5	3.0	3.2	3.2	3.4	3.1	
4			HNO ₃ /HClO ₄ -FAAS						
	7.5	24.8	5.7	6.5	6.5	10.6	6.8	8.9	
	4.7 ^{RT}	8.5	4.2	4.4	5.1	5.0	4.3	4.9	
	5.4 ^{RT}	11.1	5.1	4.5	5.6	5.7	6.3	5.4	
5			HNO ₃ -HClO ₄ -FAAS						
	5.0	12.6	5	4	6	5	5	5	
	-----	-----	Less than 1						
	1.5 ^{RT}	36.5	2	2	1	1	1	2	
6			HNO ₃ /HClO ₄ /Mg(NO ₃) ₂ -HCl-FAAS						
	5.6	16.9	5.0	7.3	4.8	6.2	5.1	5.4	
	1.3	10.9	1.1	1.3	1.5	1.3	1.2	1.4	
	2.8	19.5	2.6	3.2	2.5	2.4	2.5	3.8	
7			HNO ₃ -HClO ₄ -FAAS						
	7.1	9.1	8.24	6.96	6.38	6.66	6.92	7.26	
	3.1 ^{RT}	7.4	3.09	3.16	3.52	2.88	3.20	2.92	
	4.1 ^{RT}	8.1	4.11	4.41	4.27	4.42	3.70	3.70	
8			HNO ₃ /HClO ₄ -ICP						
	5.6	5.8	5.94	4.99	5.45	5.73	5.67	5.55	
	1.7	1.3	1.64	1.69	1.66	1.63	1.64	1.64	
	2.4	1.9	2.51	2.42	2.45	2.44	2.47	2.37	
9			HNO ₃ -GFAAS						
	5.9	4.0	5.64	5.95	5.83	6.21			
	1.2	6.1	1.19	1.22	1.19	1.26	1.05	1.16	
	2.6	8.9	2.37	2.71	2.62	2.96	2.64	2.33	
10			HNO ₃ /H ₂ SO ₄ -H ₂ O ₂ -FAAS						
	5.7	24.1	6	6	6	6	7	3	
	-----	-----	Less than 2						
	-----	-----	Less than 2						
11			NO DATA						
12			DRY ASH-HNO ₃ /HCl/HClO ₄ -FAAS						
	7.2	5.7	7	7	8	7	7	7	
	1.4	39.1	2	1	1	2	1		
	4.0 ^{RT}	22.4	5	5	4	4	3	3	

COPPER DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
13			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{H}_2\text{O}_2$ -FAAS					
	5.5	3.3	5.7	5.6	5.3	5.3	5.7	5.5
	1.9	8.4	1.2	1.3	1.3	1.1	1.2	1.4
	2.1	8.2	2.4	2.0	2.3	2.1	2.0	2.0
14			HNO_3 -DRY ASH- HNO_3 -ICP					
	6.1	14.3	5.0	6.6	5.4	5.5	6.6	7.2
	1.1	11.8	1.2	1.3	1.0	1.0	1.2	
	2.5	4.1	2.5	2.5	2.4	2.7	2.6	2.5
15			HNO_3 -DRY ASH- HNO_3 -ICP					
	5.5	17.4	5.8	7.0	4.2	5.2	4.9	5.6
	1.4	31.3	1.1	1.4	1.1	2.2	1.3	1.1
	2.5	9.9	2.8	2.1	2.5	2.6	2.4	2.3
16			HNO_3 -FAAS					
	4.4	8.5	3.9	4.8	4.2	4.7	4.3	
	0.9	33.2	1.3	1.0	0.5	1.0	0.7	0.7
	1.9	6.9	1.8	1.8	2.0	2.0	2.1	1.8
17			$\text{HNO}_3-\text{H}_2\text{O}_2-\text{HNO}_3$ -GFAAS					
	0.4 ^{IT}	20.9	0.48	0.36	0.30	0.54	0.38	0.44
	----	----	Less than 0.1					
	0.2 ^{IT}	35.6	0.21	0.20	0.20	0.18	0.41	0.24
18			$\text{HNO}_3-\text{HClO}_4$ -FAAS					
	6.0	6.0	5.41	5.95	6.28	5.95	6.39	6.28
	2.1 ^{IT}	8.3	2.25	2.25	1.93	1.93	2.24	1.93
	3.2	7.7	3.08	2.97	3.61	3.19	3.08	3.46
19A			PLASMA ASH-HCl-ICP					
	4.4	26.6	3.6	3.0	4.1	6.4	4.5	4.5
	----	----	Less than 0.4					
	2.2	39.6	3.9	1.8	1.8	1.9	1.5	2.3
19B			NO DATA					
20			$\text{HNO}_3-\text{HClO}_4$ -FAAS					
	6.0	18.8	4.68	5.51	6.17	5.99	7.76	
	1.3	31.6	1.27	1.34	1.82	0.81		
	3.0	22.4	3.46	3.68	2.31	2.55		
21			HNO_3 -DRY ASH- HNO_3 -FAAS					
	5.2	11.2	5.10	6.32	4.71	4.85	5.12	4.98
	1.4	17.0	1.50	1.34	1.89	1.26	1.26	1.32
	2.3	8.1	2.49	2.50	2.15	2.15	2.21	2.08
22			$\text{HNO}_3-\text{H}_2\text{O}_2$ -ICP					
	7.1	19.1	7.80	5.78	9.32	6.50	7.20	5.82
	1.0	14.5	0.97	0.85	1.02	0.88	1.21	
	2.8	7.2	3.01	2.54	2.65	2.80	3.00	3.01
23			HNO_3 -FAAS					
	8.3	20.4	11.2	7.44	7.17	6.51	9.09	8.21
	1.3	19.7	1.19	1.16	1.20	1.19	1.30	1.83
	2.9	8.2	2.84	2.85	2.61	2.67	3.04	3.24
24			$\text{HNO}_3-\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HNO}_3$ -FAAS					
	7.5	5.6	7.3	8.1	7.2	7.7	6.9	7.5
	7.1 ^{IT}	5.7	6.9	6.4	7.5	7.4	7.0	7.3
	4.8 ^{IT}	7.5	5.1	5.0	4.3	4.7	5.2	4.5

COPPER DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
25			$\text{HNO}_3/\text{HClO}_4/\text{H}_2\text{SO}_4$ -DCP						
	11 ^{RT}	46.2	16.4	6.3	10.3				
	1.5	13.3	1.7	1.3	1.5				
	2.6	13.3	2.8	2.8	2.2				
26			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HNO}_3-\text{H}_2\text{O}_2$ -FAAS						
	5.8	15.0	5.94	5.87	5.37	4.90	7.44	5.44	
	0.7	18.2	0.84	0.68	0.57	0.89	0.59	0.76	
	2.5	19.2	1.75	2.44	2.51	2.41	3.22	2.79	
27			AQUA REGIA- H_3BO_3 -FAAS						
	4.4	11.3	5.0	3.8	4.7	4.6	3.8	4.3	
	----	----	Less than 1.5						
	----	----	Less than 1.5						
28			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HNO}_3-\text{H}_2\text{O}_2$ -GFAAS						
	2.9 ^{RT}	20.1	1.96	3.75	3.05	2.90	2.85	2.72	
	1.1	24.9	1.29	1.11	1.61	0.95	0.88	0.99	
	2.2	49.7	3.43	3.68	1.22	1.37	1.75	1.64	
29			DRY ASH- HNO_3 -FAAS						
	----	----	Less than 8						
	----	----	Less than 1						
	----	----	Less than 3						
30			$\text{HNO}_3/\text{HClO}_4$ -ICP						
	6.4	13.2	6.1	6.4	5.6	6.0	7.8		
	2.1 ^{RT}	20.2	2.7	2.4	2.0	1.6	2.0	1.7	
	4.0 ^{RT}	16.8	3.4	3.8	3.8	3.4	5.0	4.7	
31			DRY ASH- HNO_3 -FAAS						
	6.4	12.7	6.96	5.56	6.95	7.29	6.08	5.38	
	1.6	14.5	1.36	1.53	1.70	1.36	1.87	1.87	
	2.8	7.2	2.91	2.74	3.08	2.57	2.74	2.57	
32			$\text{HNO}_3-\text{HClO}_4$ -ICP						
	5.9	13.6	6.4	4.9	5.0	6.8	6.5	6.0	
	1.1	36.6	1.2	0.65	0.76	1.5	1.6	0.85	
	2.5	21.1	3.0	2.4	2.9	2.0	1.8	3.0	
33			NO DATA						
34			NO DATA						
35			NO DATA						
36			DRY ASH- $\text{HNO}_3/\text{HClO}_4-\text{HNO}_3$ -ASV						
	6.5	10.0	5.91	7.49	6.73	5.98	6.82	5.91	
	1.3	13.4	1.36	1.47	1.18	1.10	1.46	1.11	
	2.7	10.1	2.76	2.61	2.53	3.26	2.62	2.56	
37			PLASMA ASH- $\text{HNO}_3-\text{HClO}_4$ -FAAS						
	5.2	20.6	4.40	5.69	4.09	4.75	5.10	7.03	
	0.8	36.6	0.980	1.055	0.836	0.938	0.276	0.642	
	2.8	25.8	2.03	1.99	3.74	3.42	2.94	2.59	
38			$\text{HNO}_3/\text{HClO}_4$ -GFAAS						
	7.8	7.6	7.88	7.79	6.72	7.69	8.51	8.08	
	0.2 ^{RT}	14.0	0.26	0.26	0.25	0.19	0.20	0.21	
	0.3 ^{RT}	13.9	0.32	0.34	0.30	0.31	0.29	0.22	

COPPER DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
39			HNO ₃ -FAAS						
	6.7	---	6.7						
	3.5 ⁿ	---	3.5						
	1.6 ⁿ	---	1.6						
40			HNO ₃ -HClO ₄ -ICP						
	4.2	18.1	5	4	3	4	5	4	
	----	----	Less than 1						
	2.0	31.6	1	2	2	3	2	2	
41A			FUMING HNO ₃ -HNO ₃ /H ₂ O ₂ -GFAAS						
	5.9	7.2	6.00	6.02	6.51	5.81	5.87	5.19	
	1.2	10.1	1.23	1.15	1.28	1.15	1.04	1.39	
	2.8	13.3	2.83	3.51	2.80	2.88	2.53	2.44	
41B			DRY ASH-HNO ₃ -ICP						
	5.9	6.3	5.6	5.9	5.8	5.6	6.5	6.3	
	1.2	19.1	1.00	1.43	1.06	0.99	1.53	1.23	
	2.6	10.4	2.5	2.5	3.0	2.3	2.9	2.5	
41C			HNO ₃ -EP-IDMS						
	5.1	5.4	5.64	5.08	4.99	4.99	5.04		
	0.9	3.8	0.98	0.91	0.91	0.91			
	2.7	5.2	2.83	2.69	2.55				
42			HNO ₃ -HClO ₄ /H ₂ SO ₄ -GFAAS						
	6.4	28.0	5.0	7.4	9.0	4.8	4.8	7.6	
	1.3	13.3	1.5	1.1	1.3	1.1	1.3		
	2.6	4.9	2.6	2.7	2.5	2.5	2.5	2.8	
43			HNO ₃ -H ₂ O ₂ -FAAS						
	7.5	23.5	11	7	7	7	7	6	
	1.5	36.5	2	2	1	1	2	1	
	4.0 ⁿ	57.6	3	3	3	4	3	9	

ZINC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
1			HCl-HNO ₃ -HClO ₄ /HF-HCl-FAAS						
	25.7	8.1	25	28	24				
	46.0	2.2	47	46	45				
	33.3	4.6	33	35	32				
2			HNO ₃ -HClO ₄ -FAAS						
	28.6	3.6	28.5	27	28	29	30	29	
	49.2	2.0	48	50	49	48	50	50	
	37.5	2.8	38	37	36	37	39	38	
3A			BOMB/HNO ₃ /HClO ₄ -FAAS						
	32.1	5.4	32.9	30.1	30.8	35.0	32.0	31.5	
	46.4	1.6	45.5	47.2	46.2	47.2	46.4	45.6	
	36.1	1.0	36.1	35.5	36.6	36.1	36.0	36.0	

ZINC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
3B			BOMB/HNO ₃ /HClO ₄ -ICP						
	31.3	4.3	33.0	30.8	30.8	29.1	32.0	32.0	
	46.8	2.3	45.9	47.2	46.7	48.6	45.5	46.7	
	35.7	1.1	35.5	35.4	36.2	36.2	35.3	35.6	
4			HNO ₃ /HClO ₄ -FAAS						
	28.9	10.8	28.3	32.6	26.2	26.8	26.5	33.0	
	46.0	2.0	46.7	46.7	45.4	45.6	46.8	44.6	
	35.0	1.8	35.4	35.2	35.4	35.7	34.5	34.0	
5			HNO ₃ -HClO ₄ -FAAS						
	25.3	8.2	26	23	28	27	25	23	
	36.3 ^{RT}	4.1	35	37	37	38	37	34	
	30.5	4.0	31	31	31	31	31	28	
6			HNO ₃ /HClO ₄ /Mg(NO ₃) ₂ -HCl-FAAS						
	26.2	14.4	24	30	22	27	23	31	
	39.3	4.2	39	37	40	38	41	41	
	30.0	2.1	31	30	30	30	29	30	
7			HNO ₃ -HClO ₄ -FAAS						
	28.3	18.5	28.8	26.1	38.3	23.6	25.1	27.9	
	47.8	3.6	44.8	47.4	47.5	50.0	48.1	48.7	
	30.7	5.3	33.4	29.4	29.4	30.8	29.5	31.7	
8			HNO ₃ /HClO ₄ -ICP						
	25.8	13.5	22.9	26.6	28.8	23.7	30.8	22.2	
	47.0	1.5	46.7	46.5	46.6	47.3	46.7	48.3	
	34.4	2.1	34.7	34.7	34.4	33.5	33.7	35.5	
9			HNO ₃ -FAAS						
	28.0	6.9	28.0	27.5	26.5	31.8	27.0	27.2	
	49.9	1.2	50.6	49.8	49.6	49.8	50.7	49.1	
	37.0	2.4	36.9	38.7	36.7	36.4	37.0	36.3	
10			HNO ₃ /H ₂ SO ₄ -H ₂ O ₂ -FAAS						
	25.0	12.1	21	30	25	23	25	26	
	44.2	6.8	39	47	43	44	47	45	
	34.3	9.0	29	38	33	35	35	36	
11			INAA						
	28.8	10.9	28.2	25.9	29.5	26.7	33.9		
	49.6	1.3	48.8	49.5	49.4	49.8	50.7	49.4	
	35.9	3.8	35.7	33.7	37.5	35.8	37.2	35.6	
12			DRY ASH-HNO ₃ /HCl/HClO ₄ -FAAS						
	44.7 ^{RT}	9.6	50	42	50	41	44	41	
	28.3 ^{RT}	10.1	29	26	26	26	33	30	
	33.7	2.4	35	33	33	34	33	34	
13			HNO ₃ /H ₂ SO ₄ -H ₂ O ₂ -FAAS						
	32.7	6.9	35	33	33	35	30	30	
	58.8 ^{RT}	1.7	60	60	58	58	58	59	
	39.2	4.7	37	37	40	41	41	39	
14			HNO ₃ -DRY ASH-HNO ₃ -ICP						
	27.3	8.7	27.1	28.2	30.7	23.5	28.1	26.3	
	46.1	1.3	45.3	46.4	46.8	46.4	45.5	45.9	
	33.7	2.7	33.5	33.1	32.6	34.8	34.8	33.4	

ZINC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
15			HNO_3 -DRY ASH- HNO_3 -ICP					
	26.5	19.4	34	23	30	20	24	28
	42.0	3.7	43	41	40	44	43	41
	33.0	3.8	32	35	33	32	34	32
16			HNO_3 -FAAS					
	27.7	21.7	19.9	21.6	30.9	29.3	28.4	36.0
	35.8 ^{RT}	1.8	35.2	35.4	36.1	35.0	36.2	36.6
	29.1	3.0	27.9	29.1	30.6	29.0	28.9	29.2
17			NO DATA					
18			HNO_3 - HClO_4 -FAAS					
	30.0	5.1	28.7	28.7	29.2	29.8	31.4	32.4
	51.4	1.9	50.8	51.4	49.8	51.4	52.4	52.4
	38.7	2.5	39.3	39.8	39.3	38.2	37.2	38.2
19A			PLASMA ASH-HCl-ICP					
	24.7	8.5	25.6	26.8	24.0	22.1	22.7	27.1
	49.4	5.4	50.1	46.2	48.6	54.1	48.8	48.3
	36.5	4.4	39.6	35.5	35.9	36.5	35.1	36.1
19B			NO DATA					
20			HNO_3 - HClO_4 -FAAS					
	31.0	11.3	27.0	30.7	36.7	30.6	30.2	
	47.0	1.5	47.2	47.7	47.6	46.2	46.4	
	36.8	2.0	38.0	36.1	36.4	36.8	36.9	
21			HNO_3 -DRY ASH- HNO_3 -FAAS					
	24.4	14.2	21.2	23.8	22.0	24.8	30.0	
	42.3	3.8	41.9	40.1	42.2	41.9	45.0	42.9
	31.1	8.9	35.3	27.5	33.0	30.0	31.3	29.4
22			HNO_3 - H_2O_2 -ICP					
	23.6	8.1	22.7	24.1	22.4	26.7	22.0	
	45.4	4.6	44.2	48.7	43.2	45.0	45.7	
	33.0	6.3	32.4	29.8	32.3	32.7	35.5	35.0
23			HNO_3 -FAAS					
	29.8	13.4	36.5	25.8	29.3	29.6	28.0	
	41.0	0.8	41.2	41.2	40.5	40.8	41.1	41.4
	33.4	1.5	33.5	33.7	33.0	33.3	32.8	34.2
24			HNO_3 - $\text{HNO}_3/\text{H}_2\text{SO}_4$ - HNO_3 -FAAS					
	37.8 ^{RT}	5.1	40.6	38.6	38.9	35.4	37.3	36.0
	46.2	11.9	48.9	49.6	54.1	43.6	39.9	41.4
	44.5 ^{RT}	4.5	46.6	42.9	47.1	43.7	44.3	42.2
25			$\text{HNO}_3/\text{HClO}_4/\text{H}_2\text{SO}_4$ -DCP					
	36.3 ^{RT}	39.8	53	27	29			
	52.3	7.7	48	53	56			
	33.3	16.5	36	37	27			
26			$\text{HNO}_3/\text{H}_2\text{SO}_4$ - HNO_3 - H_2O_2 -FAAS					
	29.4	4.9	31.1	28.7	31.0	28.4	27.6	29.3
	52.1	12.1	56.6	48.5	53.6	57.5	41.0	55.6
	33.4	11.2	26.9	34.4	31.9	33.2	36.2	37.4
27			AQUA REGIA- H_3BO_3 -FAAS					
	28.3	10.5	26.4	30.3	23.9	32.4	28.9	27.9
	44.7	1.0	44.6	44.9	44.2	44.3	45.4	44.9
	33.6	1.4	33.5	34.4	33.2	33.6	33.7	33.1

ZINC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
26			HNO ₃ /H ₂ SO ₄ -HNO ₃ -H ₂ O ₂ -FAAS					
	32.8	19.5	27.8	35.6	27.4	36.3	42.5	26.9
	47.9	9.2	51.0	55.6	45.0	44.9	45.2	45.8
	37.5	9.7	43.8	35.3	34.8	37.3	36.3	
29			DRY ASH-HNO ₃ -FAAS					
	28.9	26.1	42.3	22.9	27.0	22.6	25.7	32.9
	45.5	1.8	45.2	44.2	45.2	46.0	46.3	46.3
	34.1	2.4	34.0	34.4	34.4	32.8	33.8	35.3
30			HNO ₃ -HClO ₄ -ICP					
	27.9	9.6	27.7	25.0	27.8	26.8	32.3	
	42.3	10.3	36.4	47.7	46.3	41.3	43.0	38.8
	33.8	7.4	35.1	37.0	36.0	31.0	31.7	32.2
31			DRY ASH-HNO ₃ -FAAS					
	28.7	5.2	29.7	28.7	27.1	27.8	27.8	31.1
	42.0	9.8	44.3	44.1	44.0	43.5	33.7	42.4
	32.6	1.7	31.9	32.2	33.2	32.4	33.1	33.1
32			HNO ₃ -HClO ₄ -ICP					
	34.1 ^{RT}	19.5	29.1	41.1	37.4	25.6	41.1	30.2
	44.9	6.0	42.9	42.7	42.5	45.1	48.2	48.2
	34.6	7.9	32.1	32.8	33.7	38.1	38.0	32.9
33			NO DATA					
34			INAA					
	27.7	15.8	32.5	24.4	26.2	27.0	33.6	22.7
	45.6	3.4	44.1	47.5	46.2	47.1	44.4	44.2
	33.4	4.0	32.2	33.6	33.6	32.8	32.4	35.9
35			NO DATA					
36			DRY ASH-HNO ₃ /HClO ₄ -HNO ₃ -ASV					
	25.0	4.1	25.0	25.0	23.2	26.4	25.2	24.9
	44.6	4.6	48.4	42.9	43.3	43.6	44.0	45.6
	31.2	4.9	32.3	30.7	29.1	31.3	30.4	33.5
37			PLASMA ASH-HNO ₃ -HClO ₄ -FAAS					
	25.6	8.5	23.7	27.6	26.2	22.3	26.1	27.7
	43.6	2.7	43.2	45.6	42.0	43.3	44.0	43.5
	33.9	2.9	31.9	33.9	34.5	34.1	34.1	34.6
38			HNO ₃ /HClO ₄ -GFAAS					
	29.5	17.4	27.5	35.5	25.0	36.3	25.0	27.5
	47.1	4.0	45.0	47.5	47.5	47.5	50.0	45.0
	31.6	4.0	32.5	32.5	31.5	32.8	29.5	30.8
39			HNO ₃ -FAAS					
	68.4 ^{RT}	---	68.4					
	106 ^{RT}	---	106					
	99.0 ^{RT}	---	98.8					
40			HNO ₃ -HClO ₄ -ICP					
	28.3	9.9	26	24	31	29	31	29
	44.7	2.3	46	44	45	43	45	45
	31.3	3.9	31	33	32	32	30	30
41A			FUMING HNO ₃ -HNO ₃ /H ₂ O ₂ -GFAAS					
	24.8	10.5	26.8	24.4	28.5	25.1	21.7	22.3
	44.7	2.7	44.8	46.9	43.2	44.3	44.6	44.8
	33.3	3.5	33.7	33.4	34.4	34.3	31.3	32.8

ZINC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
41B			DRY ASH-HNO ₃ -ICP						
	28.5	8.2	29.4	26.9	28.4	28.1	32.6	25.8	
	46.5	1.7	46.7	45.0	44.6	46.1	45.3	45.3	
	36.4	2.8	35.8	37.2	37.7	36.2	34.8	36.5	
41C			HNO ₃ -EP-IDMS						
	27.9	7.0	24.7	30.2	26.6	28.1	28.4	29.2	
	42.9	1.3	42.4	42.9	43.5				
	34.1	4.8	31.8	35.2	35.3	34.2			
42			HNO ₃ -HClO ₄ /H ₂ SO ₄ -FAAS						
	27.0	11.4	28	25	25	25	32		
	43.3	1.2	43	44	43	43	44	43	
	32.3	2.5	33	33	32	32	31	33	
43			HNO ₃ -H ₂ O ₂ -FAAS						
	28.5	58.7	15	18	50	50	19	19	
	27.8 ^{RT}	8.6	28	28	28	26	32	25	
	23.5 ^{RT}	2.3	24	24	23	23	23	24	

ARSENIC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
1			HCl-SnCl ₂ -HG-AgDDC						
	0.3 ^{RT}	34.6	0.4	0.4	0.2				
	0.7 ^{RT}	34.3	0.7	0.5	1.0				
	0.8 ^{RT}	12.5	0.8	0.7	0.9				
2			HNO ₃ -HClO ₄ -HG-AAS						
	26.2	8.3	24.6	26.0	23.8	30.0	27.0	25.8	
	4.6	3.2	4.7	4.4	4.7	4.7	4.9	4.6	
	3.1	5.3	3.1	3.1	3.1	3.3	2.8	3.0	
3A			INAA						
	35.1	5.9	33.2	34.7	37.7	32.5	35.7	37.0	
	6.6	6.4	5.8	6.9	6.6	6.9	6.9	6.6	
	4.6	8.7	4.3	4.8	4.7	4.0	5.0	5.0	
3B			BOMB/HNO ₃ /HClO ₄ -ICP						
	33.5	2.2	33.8	34.3	32.8	33.8	33.8	32.4	
	6.8	3.5	6.9	7.2	6.9	6.7	6.5	6.7	
	4.9	3.8	4.7	5.0	4.7	5.1	4.7	4.7	
4			NO DATA						
5			HNO ₃ -HClO ₄ -HG-AAS						
	23.2	8.2	22.5	22.0	20.5	23.5	25.5	25.0	
	4.2	10.7	4.5	4.9	4.1	3.9	3.8	3.8	
	2.5	13.4	2.2	2.2	2.9	2.9	2.3	2.7	
6			HNO ₃ -Ni-GFAAS						
	32.8	6.8	34	30	31	36	32	34	
	5.6	22.0	6.4	6.9	4.5	4.6			
	3.9	12.8	3.9	4.4	3.4				

ARSENIC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
7			NO DATA						
8			NO DATA						
9			$\text{HNO}_3/\text{V}_2\text{O}_5-\text{H}_2\text{SO}_4/\text{HNO}_3-\text{HG-AgDDC}$						
	31.6	8.6	35.5	30.7	27.3	32.3	33.0	31.0	
	5.7	6.4	5.25	6.14	5.82	5.70			
	5.3	9.9	5.88	4.69	4.65	5.58	5.67	5.55	
10			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HClO}_4-\text{HG-ICP}$						
	32.9	5.8	30.5	31.1	33.9	34.5	32.3	35.2	
	6.5	4.5	6.6	6.7	6.0	6.7	6.5		
	4.1	9.4	4.4	3.8	3.5	4.3	4.2	4.5	
11			INAA						
	38.5	6.8	36.1	40.8	35.6	38.1	42.3	38.3	
	5.8	3.4	5.66	5.68	6.14	5.65	5.89	5.68	
	3.8	1.6	3.84	3.68	3.79	3.69	3.77	3.76	
12			NO DATA						
13			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{H}_2\text{O}_2-\text{HG-AAS}$						
	4.6 ^{RT}	4.6	4.81	4.63	4.8	4.31	4.38	4.69	
	0.6 ^{RT}	16.4	0.656	0.563	0.763	0.788	0.532	0.594	
	0.4 ^{RT}	5.3	0.413	0.407	0.375	0.419	0.438	0.394	
14			$\text{HNO}_3/\text{Mg}(\text{NO}_3)_2-\text{DRY ASH-HG-AAS}$						
	35.5	2.1	35.0	36.1	36.7	35.0	35.0	35.0	
	6.7	0.0	6.7	6.7	6.7	6.7	6.7	6.7	
	4.6	2.4	4.5	4.5	4.7	4.5	4.7	4.7	
15			NO DATA						
16			NO DATA						
17			NO DATA						
18			$\text{HNO}_3-\text{HClO}_4-\text{HG-AAS}$						
	27.3	16.4	28.1	25.4	32.5	22.7	32.5	22.7	
	4.7	6.7	5.14	4.71	4.49	4.49	5.14	4.49	
	3.7	15.6	3.21	4.71	3.40	3.72	3.19	3.93	
19A			PLASMA ASH-HCl-ICP						
	34.5	4.8	34	35	32	35	34	37	
	7.3	6.9	7	7	7	8			
	6.0	19.2	5	7	5	7			
19B			NO DATA						
20			NO DATA						
21			NO DATA						
22			NO DATA						
23			NO DATA						
24			NO DATA						
25			$\text{HNO}_3/\text{HClO}_4/\text{H}_2\text{SO}_4-\text{HG-AAS}$						
	10.3 ^{RT}	64.4	18	7	6				
	0.2 ^{RT}	0.0	0.2	0.2	0.2				
	2.1	40.7	2.2	1.2	2.9				
26			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HNO}_3-\text{H}_2\text{O}_2-\text{HG-AAS}$						
	0.4 ^{RT}	8.1	0.435	0.513	0.408	0.427	0.446	0.442	
	0.2 ^{RT}	38.8	0.042	0.155	0.183	0.173	0.153	0.218	
	0.2 ^{RT}	17.8	0.186	0.165	0.106	0.154	0.139	0.155	

ARSENIC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
27			NO DATA					
28			$\text{HNO}_3/\text{H}_2\text{SO}_4-\text{HNO}_3-\text{H}_2\text{O}_2-\text{HG-AgDDC}$					
	----	----	Less than 0.1					
	----	----	Less than 0.1					
	----	----	Less than 0.1					
29			NO DATA					
30			NO DATA					
31			NO DATA					
32			$\text{HNO}_3-\text{HClO}_4-\text{ICP}$					
	30.0	0.0	30	30	30	30	30	30
	----	----	Less than 10					
	----	----	Less than 10					
33			$\text{HNO}_3/\text{HClO}_4/\text{H}_2\text{SO}_4-\text{HCl-HG-AAS}$					
	28.1	7.3	25.6	28.1	30.8	29.4	26.8	
	4.7	22.4	5.37	5.24	2.88	4.88	5.37	
	3.1	3.0	3.22	3.22	3.03	3.03	3.12	
34			INAA					
	33.8	4.5	32.0	31.8	34.6	34.5	34.0	35.6
	6.4	7.2	6.71	6.74	6.20	6.83	5.60	6.37
	4.2	2.6	4.23	4.02	4.32	4.12	4.12	4.24
35			$\text{HNO}_3-\text{HClO}_4(\text{D/E})-\text{DRY ASH/Mg}(\text{NO}_3)_2$					
			-COPRECIP(F)-GFAAS					
	24.9	3.1	24.0	25.6	26.0	25.0	24.3	24.6
	5.0	26.9	2.57	5.58	5.59	5.52	5.54	
	3.7	6.4	3.83	3.92	3.80	3.38	3.49	
36			NO DATA					
37			NO DATA					
38			NO DATA					
39			$\text{H}_2\text{SO}_4-\text{HNO}_3-\text{HG-AAS}$					
	113 ^{IT}	---	113					
	36.9 ^{IT}	---	36.9					
	----	----	Less than 1.5					
40			$\text{HNO}_3-\text{HClO}_4-\text{ICP(D)}-\text{HG-AAS(E/F)}$					
	33.2	3.5	33	35	32	33	34	32
	----	----	Less than 0.1					
	----	----	Less than 0.1					
41A			FLUMING $\text{HNO}_3-\text{HNO}_3/\text{H}_2\text{O}_2-\text{GFAAS}$					
	32.1	4.7	30.6	29.9	33.1	33.1	32.1	33.6
	5.8	12.0	5.63	4.90	7.02	6.04	5.63	5.67
	3.6	9.4	3.30	3.29	3.40	3.81	3.69	4.15
41B			$\text{HNO}_3/\text{HClO}_4/\text{H}_2\text{SO}_4-\text{HG-AAS}$					
	25.5	10.2	29	26	23	26	22	27
	6.5	7.0	6.6	6.1	5.9	6.9	6.6	7.1
	3.8	7.3	4.2	4.0	3.8	3.8	3.5	3.5
41C			NO DATA					

ARSENIC DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
42			$\text{HNO}_3\text{-HClO}_4\text{/H}_2\text{SO}_4\text{-HG-AAS}$					
	26.9	17.4	29.4	31.0	30.1	28.8	21.0	20.8
	7.0	7.1	6.5	6.5	6.7	7.7	7.0	7.4
	5.1	11.2	5.8	4.3	5.0	4.9	5.7	4.8
43			NO DATA					

CADMIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
1			$\text{HCl-HNO}_3\text{-HClO}_4\text{/HF-HCl-FAAS}$					
	----	----	Less than 0.5					
	----	----	Less than 0.5					
	----	----	Less than 0.5					
2			$\text{HNO}_3\text{-HClO}_4\text{-GFAAS}$					
	0.11	18.8	0.10	0.10	0.15	0.10	0.10	0.10
	0.80 ^{RT}	18.3	0.90	0.90	0.85	0.90	0.64	0.58
	0.40	10.0	0.40	0.40	0.45	0.45	0.36	0.36
3A			$\text{BOMB/HNO}_3\text{/HClO}_4\text{-GFAAS}$					
	0.11	8.1	0.12	0.11	0.12	0.10	0.11	0.10
	0.49	5.1	0.45	0.47	0.49	0.50	0.50	0.52
	0.26	6.9	0.31	0.34	0.30	0.29	0.30	0.34
3B			$\text{BOMB/HNO}_3\text{/HClO}_4\text{-ICP}$					
	----	----	No data					
	0.50	10.8	0.49	0.58	0.43	0.46	0.53	0.53
	----	----	No data					
4			NO DATA					
5			$\text{HNO}_3\text{-HClO}_4\text{-FAAS}$					
	0.10	0.0	0.1	0.1	0.1	0.1	0.1	0.1
	0.60	14.9	0.5	0.7	0.5	0.6	0.6	0.7
	0.43	11.9	0.4	0.5	0.4	0.4	0.5	0.4
6			$\text{HNO}_3\text{/HClO}_4\text{/Mg(NO}_3)_2\text{-HCl-FAAS}$					
	----	----	Less than 0.05					
	0.52	9.1	0.46	0.52	0.51	0.6	0.49	0.51
	0.41	16.0	0.41	0.39	0.39	0.54	0.39	0.35
7			$\text{HNO}_3\text{-HClO}_4\text{-FAAS}$					
	----	----	Less than 1					
	----	----	Less than 1					
	----	----	Less than 1					
8			$\text{HNO}_3\text{/HClO}_4\text{-ICP}$					
	----	----	Less than 0.13					
	0.53	4.8	0.51	0.54	0.57	0.51	0.51	0.51
	0.39	11.4	0.34	0.41	0.44	0.43	0.34	0.37
9			$\text{HNO}_3\text{-GFAAS}$					
	0.08	20.5	0.07	0.07	0.08	0.11	0.07	0.07
	0.54	7.0	0.50	0.53	0.49	0.58	0.56	0.57
	0.33	2.6	0.33	0.31	0.32	0.33	0.33	0.33

CADMIUM DATA

CADMIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
23	-----	-----	$\text{HNO}_3\text{-FAAS}$					
	0.69 ^{RT}	10.6	0.65	0.70	0.59	0.60	0.78	0.69
	-----	-----	Less than 0.5					
24	-----	-----	$\text{HNO}_3\text{-HNO}_3\text{/H}_2\text{SO}_4\text{-HNO}_3\text{-FAAS}$					
	3.8 ^{RT}	11.8	4.4	3.6	3.5	4.2	3.5	3.3
	4.8 ^{RT}	8.2	4.6	4.9	5.4	4.3	4.6	5.1
	4.0 ^{RT}	5.0	4.2	4.1	4.1	3.9	3.8	3.7
25	-----	-----	$\text{HNO}_3\text{/HClO}_4\text{/H}_2\text{SO}_4\text{-DCP}$					
	0.47 ^{RT}	49.5	0.6	0.6	0.2			
	0.90 ^{RT}	11.1	0.8	0.9	1.0			
	0.40	0.0	0.4	0.4	0.4			
26	-----	-----	$\text{HNO}_3\text{/H}_2\text{SO}_4\text{-HNO}_3\text{-H}_2\text{O}_2\text{-FAAS}$					
	-----	-----	Less than 0.16					
	0.40	5.7	0.38	0.37	0.41	0.42	0.42	0.38
	0.21	12.8	0.18	0.19	0.22	0.19	0.24	0.24
27	-----	-----	AQUA REGIA- $\text{H}_3\text{BO}_3\text{-FAAS}$					
	-----	-----	Less than 0.5					
	0.88 ^{RT}	17.3	0.97	0.93	1.02	0.69	0.69	1.00
	0.75 ^{RT}	19.1	1.03	0.68	0.72	0.72	0.70	0.63
28	-----	-----	$\text{HNO}_3\text{/H}_2\text{SO}_4\text{-HNO}_3\text{-H}_2\text{O}_2\text{-GFAAS}$					
	0.04	6.6	0.046	0.045	0.040	0.040	0.044	
	0.39	21.6	0.364	0.498	0.353	0.477	0.283	0.343
	0.28	27.6	0.425	0.215	0.282	0.267	0.279	0.213
29	-----	-----	DRY ASH- $\text{HNO}_3\text{-ASV}$					
	-----	-----	Less than 0.02					
	0.41	12.4	0.43	0.39	0.33	0.45	0.45	
	0.25	9.6	0.26	0.24	0.25	0.22	0.22	0.28
30	-----	-----	$\text{HNO}_3\text{-HClO}_4\text{-GFAAS}$					
	-----	-----	Less than 0.1					
	0.37	58.9	0.3	0.3	0.2	0.3	0.3	0.8
	0.20	0.0	0.2	0.2	0.2	0.2	0.2	0.2
31	-----	-----	DRY ASH- $\text{HNO}_3\text{-FAAS}$					
	-----	-----	Less than 0.17					
	0.51	0.0	0.51	0.51	0.51	0.51	0.51	0.51
	0.34	0.0	0.34	0.34	0.34	0.34	0.34	0.34
32	-----	-----	$\text{HNO}_3\text{-HClO}_4\text{-ICP}$					
	-----	-----	Less than 0.1					
	0.50	17.9	0.4	0.4	0.5	0.6	0.5	0.6
	0.30	29.8	0.3	0.2	0.2	0.4	0.3	0.4
33	-----	-----	NO DATA					
34	-----	-----	$\text{HNO}_3\text{-GFAAS}$					
	0.08	12.3	0.078	0.087	0.073	0.066	0.077	0.093
	0.52	6.4	0.52	0.52	0.56	0.46	0.54	0.52
	0.33	8.0	0.35	0.31	0.37	0.32	0.32	0.30
35	-----	-----	NO DATA					
36	-----	-----	DRY ASH- $\text{HNO}_3\text{/HClO}_4\text{/HNO}_3\text{-ASV}$					
	0.17 ^{RT}	3.6	0.174	0.174	0.164	0.161	0.164	0.162
	0.48	8.9	0.521	0.438	0.451	0.531	0.485	0.434
	0.28	4.1	0.297	0.278	0.262	0.275	0.276	0.280

CADMIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
37			PLASMA ASH-HNO ₃ -HClO ₄ -GFAAS					
	0.09	18.7	0.072	0.083	0.077	0.111	0.106	0.108
	0.46	5.8	0.459	0.449	0.489	0.442	0.495	0.427
	0.27	7.7	0.269	0.257	0.304	0.296	0.258	0.257
38			HNO ₃ /HClO ₄ -GFAAS					
	0.07	18.2	0.06	0.06	0.06	0.07	0.09	0.06
	0.44	11.7	0.37	0.43	0.45	0.52	0.45	0.40
	0.27	4.1	0.27	0.29	0.27	0.27	0.26	0.26
39			HNO ₃ -FAAS					
	----	----	Less than 0.07					
	----	----	Less than 0.07					
	0.45 ^{RT}	----	0.45					
40			HNO ₃ -HClO ₄ -ICP					
	----	----	Less than 1					
	----	----	Less than 1					
	----	----	Less than 1					
41A			FUMING HNO ₃ -HNO ₃ /H ₂ O ₂ -GFAAS					
	0.06	28.3	0.064	0.058	0.084	0.046	0.046	0.041
	0.43	6.0	0.458	0.417	0.453	0.389	0.424	0.443
	0.28	7.2	0.310	0.300	0.293	0.264	0.263	0.269
41B			DRY ASH-HNO ₃ -ICP					
	0.22 ^{RT}	27.6	0.16	0.21	0.19	0.21	0.19	0.33
	0.52	11.0	0.47	0.59	0.49	0.56	0.56	0.45
	0.36	20.0	0.37	0.32	0.48	0.32	0.41	0.28
41C			HNO ₃ -EP-IDMS					
	0.07	11.9	0.060	0.077	0.062	0.077	0.079	0.077
	0.57	17.5	0.597	0.485	0.488	0.691		
	0.34	7.5	0.300	0.355	0.347	0.349		
42			HNO ₃ -HClO ₄ /H ₂ SO ₄ -GFAAS					
	0.25 ^{RT}	19.2	0.23	0.25	0.31	0.25	0.23	0.23
	0.76 ^{RT}	6.9	0.85	0.73	0.75	0.79	0.75	0.70
	0.53 ^{RT}	7.4	0.52	0.50	0.54	0.48	0.59	0.55
43			HNO ₃ -H ₂ O ₂ -FAAS					
	----	----	Less than 0.1					
	0.53	38.4	0.29	0.49	0.51	0.89	0.60	0.41
	0.35	13.1	0.28	0.34	0.35	0.42	0.36	0.33

MERCURY DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
1			HNO ₃ /HCl-SnSO ₄ -CVAAS					
	1.35	1.7	1.36	1.34	1.32	1.38	1.34	
	0.106	0.0	0.106	0.106	0.106	0.106	0.106	
	3.00	0.0	3.00	3.00	3.00	3.00	3.00	

MERCURY DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
2			$\text{HNO}_3\text{-HClO}_4\text{-SnCl}_2\text{-CVAAS}$					
	1.72	12.1	2.00	1.70	1.80	1.90	1.42	1.60
	0.050	0.0	0.05	0.05	0.05			
	4.00	11.2	4.80	4.40	3.90	3.60	3.80	3.80
3A			$\text{HNO}_3\text{/K}_2\text{S}_2\text{O}_8\text{-SnCl}_2\text{-CVAAS}$					
	2.11	3.2	2.06	2.11	2.05	2.13	2.06	2.23
	0.067	7.0	0.069	0.069	0.073	0.061	0.062	0.065
	4.83	0.7	4.83	4.85	4.79	4.86	4.78	4.86
3B			NO DATA					
4			NO DATA					
5			$\text{HNO}_3\text{/H}_2\text{SO}_4\text{/K}_2\text{S}_2\text{O}_8\text{/KMnO}_4\text{-NH}_2\text{OH}\text{-HCl-SnSO}_4\text{-CVAAS}$					
	2.10	9.4	1.80	2.00	2.25	2.30	2.00	2.25
	0.038	13.7	0.030	0.030	0.040	0.040	0.035	0.040
	4.30	6.5	4.60	4.10	4.20	3.90	4.35	4.60
6			$\text{HNO}_3\text{/H}_2\text{SO}_4\text{-KMnO}_4\text{-NH}_2\text{OH}\text{-HCl-SnSO}_4\text{-CVAAS}$					
	1.68	8.4	1.4	1.6	1.8	1.7	1.6	1.7
	0.025	16.6	0.022	0.024	0.025	0.026	0.020	0.032
	3.88	3.8	3.8	3.8	4.1	3.7	3.9	4.0
7			$\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-KMnO}_4\text{-CVAAS}$					
	1.84	8.2	1.94	1.78	1.60	1.81	2.04	1.85
	----	----	Less than 0.14					
	5.91	6.0	6.59	5.65	5.62	5.81	5.94	5.86
8			$\text{HNO}_3\text{/H}_2\text{SO}_4\text{/HClO}_4\text{-SnCl}_2\text{-CVAAS}$					
	1.48	12.4	1.12	1.51	1.51	1.57	1.54	1.64
	0.061	21.6	0.075	0.058	0.041	0.071	0.062	
	2.12 ⁿ	16.5	2.18	2.45	2.61	1.84	1.78	1.86
9			$\text{HNO}_3\text{/H}_2\text{SO}_4\text{-KMnO}_4\text{-NH}_2\text{OH}\text{-HCl-SnCl}_2\text{-CVAAS}$					
	1.60	13.4	1.45	1.95	1.36	1.68	1.49	1.69
	0.045	15.0	0.032	0.046	0.044	0.051	0.045	0.049
	2.54 ⁿ	1.8	2.57	2.57	2.53	2.59	2.48	2.49
10			$\text{HNO}_3\text{/H}_2\text{SO}_4\text{-KMnO}_4\text{-K}_2\text{S}_2\text{O}_8\text{-NH}_2\text{OH-SnCl}_2\text{-CVAAS}$					
	2.03	2.5	2.0	2.1	2.1	2.0	2.0	2.0
	----	----	Less than 0.1					
	4.95	4.7	5.0	4.5	4.9	5.1	5.1	5.1
11			NO DATA					
12			NO DATA					
13			$\text{H}_2\text{SO}_4\text{/K}_2\text{Cr}_2\text{O}_7\text{-CVAAS}$					
	1.58	6.2	1.7	1.7	1.6	1.5	1.5	1.5
	0.040	41.8	0.03	0.03	0.03	0.03	0.05	0.07
	3.98	1.9	3.9	3.9	4.1	4.0	4.0	4.0
14			NO DATA					
15			NO DATA					
16			NO DATA					
17			NO DATA					
18			$\text{HNO}_3\text{-HClO}_4\text{-CVAAS}$					
	1.87	5.4	1.89	1.84	1.70	2.00	1.87	1.93
	0.029	18.0	0.022	0.022	0.032	0.032	0.032	0.032
	3.83	9.7	3.66	3.29	4.25	3.82	4.25	3.72

MERCURY DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
19A			$H_2SO_4-H_2O_2-HNO_3-NH_2OH-SnCl_2-CVAAS$					
	1.35	21.4	1.68	1.48	0.88	1.42	1.14	1.50
	0.123	8.4	0.13	0.11	0.11	0.13	0.13	0.13
	3.83	13.3	4.4	4.4	3.3	3.3	4.0	3.6
19B			NO DATA					
20			$HNO_3/HCl-K_2S_2O_8-CVAAS$					
	1.90	4.8	1.79	1.82	1.92	1.93	2.05	1.91
	0.045	46.1	0.04	0.05	0.02	0.08	0.05	0.03
	5.06	2.2	4.92	4.92	5.14	5.17	5.12	5.11
21			$HNO_3/H_2SO_4-KMnO_4-CVAAS$					
	2.08	10.9	2.31	1.94	2.29	2.26	1.82	1.88
	----	----	Less than 0.01					
	4.82	4.6	4.50	4.79	4.83	4.81	4.78	5.20
22			NO DATA					
23			NO DATA					
24			NO DATA					
25			NO DATA					
26			$H_2SO_4-CVAAS$					
	2.11	11.3	2.02	1.80	2.09	2.25	1.99	2.49
	----	----	Less than 0.03					
	3.77	13.0	2.96	4.45	3.72	3.97	3.61	3.93
27			NO DATA					
28			NO DATA					
29			$HNO_3/H_2SO_4-CVAAS$					
	2.07	9.0	2.19	2.15	1.85			
	----	----	No data					
	4.65	5.0	4.77	4.80	4.38			
30			NO DATA					
31			NO DATA					
32			HNO_3-HClO_4-ICP					
	----	----	Less than 4					
	----	----	Less than 4					
	----	----	Less than 4					
33			NO DATA					
34			NO DATA					
35			NO DATA					
36			NO DATA					
37			NO DATA					
38			NO DATA					
39			$H_2SO_4-HNO_3-NaBH_4-CVAAS$					
	1.04 ^{IT}	----	1.04					
	----	----	Less than 0.02					
	1.47 ^{IT}	----	1.47					
40			$H_2SO_4/HNO_3-KMnO_4/K_2S_2O_8-NH_2OH-SnCl_2-CVAAS$					
	1.96	3.9	2.05	2.06	1.93	1.90	1.88	1.93
	0.070	15.6	0.06	0.06	0.06	0.08	0.08	0.08
	4.73	1.1	4.68	4.71	4.76	4.76	4.80	4.67

MERCURY DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
41A			HNO ₃ /H ₂ SO ₄ -NaBH ₄ -CVAAS						
	2.17	6.9	2.3	2.1	2.0	2.1	2.4	2.1	
	0.029	21.5	0.032	0.029	0.023	0.022	0.029	0.039	
	4.37	2.4	4.4	4.3	4.4	4.2	4.4	4.5	
41B			NO DATA						
41C			NO DATA						
42			HNO ₃ /H ₂ SO ₄ -KMnO ₄ /K ₂ S ₂ O ₈ -NH ₂ OH-SnCl ₂ -CVAAS						
	2.00	8.9	2.04	1.93	2.28	1.78	1.87	2.09	
	0.028	40.5	0.034	0.023	0.018	0.020	0.045		
	4.54	12.3	4.97	3.58	4.88	4.68	4.60		
43			NO DATA						

LEAD DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
1			HCl-HNO ₃ -HClO ₄ /HF-HCl-FAAS						
	2.0 ⁿ	0.0	2	2	2				
	----	----	Less than 2						
	----	----	Less than 2						
2			HNO ₃ -HClO ₄ -GFAAS						
	9.9	2.1	10.0	10.0	10.0	10.0	10.0	9.5	
	----	----	Less than 0.1						
	0.22	11.9	0.20	0.25	0.20	0.25	0.20	0.20	
3A			BOMB/HNO ₃ /HClO ₄ -GFAAS						
	5.1	4.8	5.1	5.2	5.2	4.7	5.3	4.8	
	0.10	17.9	0.08	0.09	0.09	0.13	0.11	0.10	
	0.30	14.1	0.35	0.32	0.33	0.25	0.25	0.32	
3B			BOMB/HNO ₃ /HClO ₄ -ICP						
	5.0	17.0	6.0	5.4	5.8	4.0	4.6	4.2	
	----	----	Less than 3						
	----	----	Less than 3						
4			NO DATA						
5			HNO ₃ -HClO ₄ -FAAS						
	10.3	7.9	11	10	10	11	11	9	
	----	----	Less than 1						
	----	----	Less than 1						
6			HNO ₃ /HClO ₄ /Mg(NO ₃) ₂ -HCl-FAAS						
	9.9	17.8	8.8	11	9.2	9.0	13	8.4	
	----	----	Less than 1						
	----	----	Less than 1						
7			HNO ₃ -HClO ₄ -FAAS						
	12.1	10.4	11.0	11.8	13.5	11.3	11.2	13.9	
	6.22 ⁿ	12.8	5.41	6.08	5.86	7.69	6.41	5.84	
	5.51 ⁿ	13.3	5.28	4.90	6.64	5.77	4.62	5.87	

LEAD DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
8			$\text{HNO}_3/\text{HClO}_4\text{-ICP}$						
	10.0	9.5	9.27	10.2	10.6	11.4	9.30	8.93	
	----	----	Less than 2						
	----	----	Less than 2						
9			$\text{HNO}_3\text{-GFAAS}$						
	11.2	7.1	10.1	11.8	10.6	11.0	11.3	12.3	
	----	----	Less than 0.5						
	----	----	Less than 0.5						
10			$\text{HNO}_3/\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-EXTN-FAAS}$						
	7.8	9.6	8	9	8	7	7	8	
	----	----	Less than 1						
	----	----	Less than 1						
11			NO DATA						
12			DRY ASH- $\text{HNO}_3/\text{HCl/HClO}_4\text{-FAAS}$						
	11.2	6.7	11	10	11	11	12	12	
	4.5 ^{RT}	12.2	5	5	4	5	4	4	
	5.0 ^{RT}	20.3	5	6	6	4	4	4	
13			$\text{HNO}_3/\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-EXTN-FAAS}$						
	6.2	5.3	6.3	6.3	6.3	6.3	6.6	5.6	
	0.18	14.4	0.186	0.188	0.188	0.188	0.188	0.125	
	0.34	9.6	0.369	0.313	0.313	0.313	0.375	0.375	
14			$\text{HNO}_3\text{-DRY ASH-HNO}_3\text{-ICP}$						
	9.7	12.7	12.1	8.8	9.0	9.2	9.4	9.4	
	----	----	Less than 0.8						
	----	----	Less than 0.8						
15			$\text{HNO}_3\text{-DRY ASH-HNO}_3\text{-ICP}$						
	7.7	10.4	8.8	6.7	7.4	8.1	7.4		
	----	----	Less than 0.2						
	2.53 ^{RT}	2.0	2.5	2.6	2.5	2.5	2.5	2.6	
16			NO DATA						
17			NO DATA						
18			$\text{HNO}_3/\text{HClO}_4\text{-GFAAS}$						
	10.0	11.2	10.8	8.93	11.9	9.52	9.31	9.53	
	0.26	19.5	0.27	0.27	0.21	0.20	0.32	0.32	
	0.14	15.6	0.16	0.16	0.16	0.12	0.12	0.12	
19A			PLASMA ASH-HCl-ICP						
	8.3	19.6	8	8	6	8	9	11	
	----	----	Less than 2						
	----	----	Less than 2						
19B			PLASMA ASH-HCl-GFAAS						
	9.0	18.1	7.8	8.1	7.7	8.6	10.2	11.8	
	0.15	73.5	0.09	0.09	0.10	0.31			
	0.16	8.7	0.16	0.15	0.14	0.16	0.15	0.18	
20			$\text{HNO}_3\text{-HClO}_4\text{-EXTN-FAAS}$						
	9.8	6.8	9.54	10.4	9.04	10.5	9.29		
	0.19	38.0	0.27	0.17	0.13				
	0.25	43.3	0.13	0.28	0.34				

LEAD DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
21			HNO ₃ -DRY ASH-HNO ₃ -FAAS					
	8.3	6.9	8.00	8.21	9.13	8.02	7.51	8.69
	----	----	Less than 0.2					
	----	----	Less than 0.2					
22			HNO ₃ -H ₂ O ₂ -ICP					
	10.4	11.1	10.2	10.3	10.5	8.4	11.0	11.9
	----	----	Less than 5					
	----	----	Less than 4					
23			HNO ₃ -FAAS					
	11.0	19.0	10.5	9.5	14.7	10.4	10.0	
	1.70 ^{RT}	7.4	1.7	1.5	1.7	1.7	1.9	1.7
	1.22 ^{RT}	15.1	1.2	1.4	1.4	1.0	1.3	1.0
24			HNO ₃ -HNO ₃ /H ₂ SO ₄ -HNO ₃ -FAAS					
	66 ^{RT}	7.2	73.5	63.8	66.3	69.9	60.7	63.1
	61 ^{RT}	4.9	57.4	61.7	64.6	57.9	60.6	63.8
	55 ^{RT}	6.6	56.4	60.5	53.4	53.0	56.8	50.2
25			HNO ₃ /HClO ₄ /H ₂ SO ₄ -DCP					
	5.0	30.3	6.7	3.7	4.7			
	1.37 ^{RT}	33.0	0.9	1.4	1.8			
	0.25	----	0.2	0.3				
26			HNO ₃ /H ₂ SO ₄ -HNO ₃ -H ₂ O ₂ -FAAS					
	12.7	16.3	10.4	11.2	15.0	11.4	15.3	12.8
	3.12 ^{RT}	14.0	3.04	2.72	2.62	3.56	3.06	3.70
	2.63 ^{RT}	7.9	2.51	2.77	2.32	2.89	2.55	2.74
27			AQUA REGIA-H ₃ BO ₃ -FAAS					
	----	----	Less than 10					
	----	----	Less than 10					
	----	----	Less than 10					
28			HNO ₃ /H ₂ SO ₄ -HNO ₃ -H ₂ O ₂ -GFAAS					
	0.4 ^{RT}	16.3	0.41	0.43	0.40	0.33	0.46	0.54
	----	----	Less than 0.05					
	----	----	Less than 0.05					
29			DRY ASH-HNO ₃ -ASV					
	12.3	21.6	15.5	10.5	13.8	12.8	8.80	
	1.82 ^{RT}	22.8	2.2	2	2.1	1.2	1.6	
	1.88 ^{RT}	27.2	1.7	2.6	1.3	1.8	1.5	2.4
30			HNO ₃ -HClO ₄ -GFAAS					
	4.9	10.2	4.6	4.3	5.4	5.1		
	0.27	30.6	0.2	0.2	0.2	0.3	0.4	0.3
	0.32	31.0	0.3	0.2	0.2	0.4	0.4	0.4
31			DRY ASH-HNO ₃ -EXTN-FAAS					
	10.2	7.0	11.1	10.1	9.1	9.7	10.3	10.7
	0.47	9.3	0.43	0.51	0.43	0.43	0.51	0.51
	0.67 ^{RT}	20.3	0.77	0.77	0.69	0.43	0.60	0.77
32			HNO ₃ -HClO ₄ -ICP					
	7.5	10.5	6.9	7.0	6.5	8.0	8.0	8.5
	----	----	Less than 3					
	----	----	Less than 3					

LEAD DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
33			NO DATA					
34			HNO ₃ -GFAAS					
	9.1	10.5	7.5	6.8	8.2	8.8	9.1	8.4
	----	----	Less than 0.1					
	0.20	5.8	0.21	0.21	0.21	0.20	0.18	0.20
35			NO DATA					
36			DRY ASH-HNO ₃ /HClO ₄ -HNO ₃ -ASV					
	9.2	8.6	9.20	8.37	9.67	8.13	9.69	10.1
	0.33	48.7	0.373	0.312	0.16	0.179	0.603	0.353
	0.27	21.5	0.351	0.267	0.258	0.205	0.205	0.304
37			PLASMA ASH-HNO ₃ -HClO ₄ -GFAAS					
	3.3 ⁿ	21.3	3.48	4.14	2.56	2.89	2.54	3.92
	----	----	Less than 0.5					
	----	----	Less than 0.5					
38			HNO ₃ /HClO ₄ -GFAAS					
	7.8	7.6	7.88	7.79	6.72	7.69	8.51	8.08
	0.23	14.0	0.26	0.26	0.25	0.19	0.20	0.21
	0.30	13.9	0.32	0.34	0.30	0.31	0.29	0.22
39			HNO ₃ -FAAS					
	6.7	----	6.7					
	3.5 ⁿ	----	3.5					
	1.6 ⁿ	----	1.6					
40			HNO ₃ -HClO ₄ -GFAAS					
	11.7	24.6	14	9	12	16	9	10
	----	----	Less than 1					
	----	----	Less than 1					
41A			FUMING HNO ₃ -HNO ₃ /H ₂ O ₂ -GFAAS					
	9.3	13.2	9.15	8.43	7.52	10.7	10.5	9.8
	0.06	11.1	0.061	0.057	0.05	0.061	0.068	
	0.19	24.5	0.177	0.131	0.259	0.187	0.182	
41B			DRY ASH-HNO ₃ -ICP					
	8.9	9.2	8.0	8.4	9.2	8.6	10.1	
	----	----	Less than 0.9					
	----	----	Less than 0.8					
41C			HNO ₃ -EP-IDMS					
	9.9	8.2	8.53	10.1	9.31	10.3	10.5	10.6
	0.10	3.8	0.100	0.103	0.103	0.095		
	0.22	5.4	0.211	0.224	0.235			
42			HNO ₃ -HClO ₄ /H ₂ SO ₄ -GFAAS					
	7.0	29.6	6.3	7.9	4.6	10.5	6.9	5.6
	----	----	Less than 0.3					
	----	----	Less than 0.3					
43			HNO ₃ -H ₂ O ₂ -FAAS					
	15.2 ⁿ	20.5	13	18	14	19	12	
	3.00 ⁿ	47.1	5	4	1	3	3	2
	1.83 ⁿ	22.3	1	2	2	2	2	

SODIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
19A			PLASMA ASH-HCl-ICP						
	0.316	2.2	0.316	0.318	0.311	0.315	0.309	0.329	
	1.18	4.2	1.18	1.13	1.15	1.27	1.18	1.15	
	0.317	1.8	0.326	0.309	0.314	0.321	0.311	0.321	
27			AQUA REGIA-H ₃ BO ₃ -FAAS						
	0.302	2.3	0.314	0.299	0.293	0.303	0.303	0.303	
	0.987	0.6	0.986	0.986	0.989	0.978	0.990	0.995	
	0.306	3.2	0.321	0.304	0.308	0.310	0.290	0.305	
34			INAA						
	0.292	0.9	0.292	0.289	0.293	0.294	0.290	0.296	
	1.10	1.6	1.08	1.11	1.10	1.13	1.09	1.09	
	0.289	2.7	0.289	0.294	0.297	0.281	0.294	0.278	

CALCIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
14			HNO ₃ -DRY ASH-HNO ₃ -ICP						
	250	2.0	252	257	249	243	243	248	
	558	3.9	534	582	534	550	583	564	
	189	6.4	171	196	192	204	187	182	
19A			PLASMA ASH-HCl-ICP						
	263	6.4	266	261	242	275	248	287	
	529	7.8	606	560	589	688	578	568	
	280	26.2	236	223	264	372	213	372	
27			AQUA REGIA-H ₃ BO ₃ -FAAS						
	253	7.1	287	239	255	250	243	241	
	529	3.0	542	513	549	534	528	510	
	223	8.7	208	201	249	238	231	209	

CHROMIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>							
14			HNO ₃ -DRY ASH-HNO ₃ -ICP						
	0.7	28.6	0.9	0.6	0.7	0.4	0.6	0.7	
	1.0	10.0	1.2	1.1	1.0	1.0	0.8	1.0	
	1.1	18.2	1.2	1.2	1.2	1.2	0.8	1.1	
17	----	----	HNO ₃ -H ₂ O ₂ -GFAAS						
			Less than 0.43						
	0.77	22.5	0.81	0.51	0.96	0.78	0.63	0.93	
	0.80	27.0	0.81	0.72	0.58	0.57	0.99	1.1	
19A			PLASMA ASH-HCl-ICP						
	0.7	33.8	1.1	0.6	0.5	0.7	0.5	0.6	
	1.1	19.8	0.8	1.2	0.9	1.1	1.4	1.1	
	1.5	19.3	1.4	1.7	1.2	1.7	1.2	1.9	

CHROMIUM DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
23			$\text{HNO}_3\text{-FAAS}$					
	1.27	8.7	1.46	1.23	1.15	1.27	1.17	1.34
	1.40	6.4	1.35	1.41	1.25	1.43	1.41	1.54
	1.10	5.5	1.06	1.21	1.07	1.08	1.13	1.02
27			$\text{AQUA REGIA-H}_3\text{BO}_3\text{-FAAS}$					
	----	----	Less than 5					
	----	----	Less than 5					
	----	----	Less than 5					
34			INAA					
	2.04	12.4	1.82	2.32	2.14	1.65	2.20	2.08
	1.00	27.6	1.02	1.28	1.17	1.17	0.82	0.54
	1.63	10.2	1.62	1.52	1.90	1.64	1.47	
41C			$\text{HNO}_3\text{-EP-IDMS}$					
	0.77	13.6	0.68	0.76	0.92	0.71		
	1.14	32.3	1.02	0.74	1.19	1.62		
	1.73	16.4	2.10	1.42	1.74	1.64		

MANGANESE DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
14			$\text{HNO}_3\text{-DRY ASH-HNO}_3\text{-ICP}$					
	3.6	8.3	3.4	4.0	3.4	4.0	3.5	3.5
	0.9	11.1	1.0	1.0	0.8	1.0	0.8	1.0
	0.5	16.0	0.4	0.5	0.4	0.6	0.5	0.4
19B			$\text{PLASMA ASH-HCl-GFAAS}$					
	4.27	18.1	4.45	5.13	3.28	3.67	3.95	5.11
	1.02	5.0	1.05	1.01	0.98	1.10	1.00	0.96
	0.57	10.7	0.68	0.52	0.56	0.57	0.51	0.59
21			$\text{HNO}_3\text{-DRY ASH-HNO}_3\text{-FAAS}$					
	2.82	11.0	2.70	2.69	2.82	2.87	2.47	3.38
	0.59	8.5	0.62	0.63	0.63	0.51	0.51	0.55
	0.37	24.3	0.30	0.34	0.34	0.55	0.34	0.34
23			$\text{HNO}_3\text{-FAAS}$					
	5.05	21.8	6.98	4.37	4.32	5.17	3.97	5.47
	1.01	2.0	0.98	1.03	0.99	1.02	1.00	1.01
	0.58	1.7	0.57	0.59	0.57	0.60	0.57	0.58

IRON DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
14			$\text{HNO}_3\text{-DRY ASH-HNO}_3\text{-ICP}$					
	123	16.3	107	130	159	114	108	122
	17.3	9.3	19.9	20.0	18.7	16.6	14.4	14.3
	29.2	30.1	24.4	45.4	23.7	33.3	23.2	25.1

IRON DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
19A			PLASMA ASH-HCl-ICP					
	121	21.7	117	158	93	106	103	148
	16.8	10.2	19.4	15.6	16.4	17.6	17.4	14.5
	27.7	9.4	29.8	26.0	31.9	26.4	25.2	26.6
23			HNO ₃ -FAAS					
	129	20.9	180	119	108	134	109	123
	15.4	2.6	14.8	15.6	15.7	15.2	15.7	15.3
	27.4	7.3	26.4	26.0	28.3	27.2	25.5	30.9
27			AQUA REGIA-H ₃ BO ₃ -FAAS					
	143	17.7	139	193	121	140	134	133
	17.0	8.4	16.3	17.9	14.8	19.0	17.0	16.8
	27.5	9.9	31.6	25.9	29.5	27.3	26.5	24.0
34			INAA					
	124	17.8	126	138	115	94	157	112
	15.6	15.4	17	19	15	14	13	
	33.4	23.0	43.3	39.2	25.7	26.0	28.1	37.8
36			DRY ASH-HNO ₃ /HClO ₄ -HNO ₃ -ASV					
	122	6.6	113	120	127	119	130	
	14.7	19.1	17.5	12.8	12.1	15.5	15.6	
	30.0	10.0	31.8	28.3	30.9	32.4	26.8	

NICKEL DATA

<u>Lab</u>	<u>Mean</u>	<u>RSD</u>						
19A			PLASMA ASH-HCl-ICP					
	----	----	Less than 2					
	----	----	Less than 2					
	----	----	Less than 2					
23			HNO ₃ -FAAS					
	1.99	13.1	2.45	1.67	1.90	2.09	1.97	1.86
	1.45	7.6	1.35	1.56	1.31	1.54	1.45	1.52
	1.47	4.8	1.43	1.49	1.51	1.40	1.40	1.59
27			AQUA REGIA-H ₃ BO ₃ -FAAS					
	----	----	Less than 5					
	----	----	Less than 5					
	----	----	Less than 5					
41C			HNO ₃ -EP-IDMS					
	1.44	22.4	1.68	1.19	1.76	1.14		
	0.56	35.0	0.37	0.46	0.58	0.82		
	0.84	2.1	0.83	0.86	0.83			
43			HNO ₃ -H ₂ O ₂ -FAAS					
	3.67	22.2	4	5	4	3	3	3
	1.67	48.9	3	1	1	1	2	2
	1.00	0.0	1	1	1	1	1	1

Mandat du Comité

Son rôle est constituer un forum qui réunit les diverses opinions sur les sujets se rapportant à la chimie marine et de les transmettre au Conseil national de recherches; de coordonner les activités de recherche en ce qui a trait aux problèmes d'analyse et à la mise au point de nouveaux standards. De façon plus précise:

- vérifier au besoin la situation de la chimie marine au Canada et en particulier l'effort national dans le domaine de la chimie analytique marine.
- coordonner la mise au point constante de standards, de méthodes d'analyse et de systèmes de comparaison entre les laboratoires.
- prévoir les besoins et les exigences à long terme du développement de la chimie analytique marine autant dans le secteur privé que public, établir les priorités et évaluer les ressources nécessaires pour répondre à ces besoins.
- favoriser les échanges entre les groupes qui étudient la mer et la chimie analytique, aider l'échange des renseignements, et proposer des méthodes d'améliorer ces échanges.
- étudier des problèmes particuliers à la demande de divers organismes qui s'intéressent à l'analyse d'échantillons marins.

- to anticipate the long term developmental needs and requirements of marine analytical chemistry in the private and public sectors, recommend priorities, and estimate the resources required to meet the needs.
- to promote communication between marine and analytical chemistry groups, assist in the exchange of information, and recommend mechanisms for improving liaison.
- to study specific problems at the request of various agencies concerned with the analysis of marine materials.

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Dr. M. B. Yunker; Dobrocky Seatech Limited, Sidney, B. C.

Terms of Reference

To provide a forum for the synthesis of advice to the National Research Council on matters pertaining to marine chemistry, and a mechanism for the coordination of research activities related to the provision of reference materials and analytical problems of marine materials. More specifically:

- to review as necessary, the status of marine chemistry in Canada and particularly the national effort in marine analytical chemistry.
- to coordinate the continuing development of standard reference materials, standard methods and interlaboratory calibrations.