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LEAD CONCENTRATE CPB-1 — A CERTIFIED REFERENCE MATERIAL

G.H. FAYE, W.S. BOWMAN AND R. SUTARNO

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LEAD CONCENTRATE CPB-1 — A CERTIFIED REFERENCE MATERIAL

by

G.H. Faye*, W.S. Bowman** and R. Sutarno**

SYNOPSIS

A 250-kg sample of a lead (flotation) concentrate, CPB-1, from Kimberley, British Columbia, has been prepared as a compositional reference material. CPB-1 was ground to minus 74 µm, mixed in one lot, tested for homogeneity by X-ray and chemical methods and bottled in 200-g units.

In a "free-choice" analytical program, 25 laboratories contributed results for one or more of 21 elements in each of two bottles of CPB-1. Based on a statistical analysis of the data, recommended values have been assigned for: Ag, Al_2O_3, As, Bi, Cd, Cu, Fe, Hg, Pb, S, Sb, SiO_2 and Zn. Also, non-certified values have been determined for: Au, CaO, In, MgO, Mn, Se, Sn and Te.

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Note: Major contributions were also made by other staff members of the Mineral Sciences Laboratories as well as of laboratories in other organizations.

CONCENTRE DE PLOMB CPB-1 - UN MATERIAU DE REFERENCE CERTIFIE

par

G.H. Faye*, W.S. Bowman** et R. Sutarno**

SOMMAIRE

Un échantillon de 250 kg de concentré de plomb (flottation), CPB-1, provenant de Kimberley en Colombie-Britannique, a été préparé en tant que matériau de référence de composition. Le CPB-1 a été broyé jusqu'à -74 µm et mélangé ensemble; l'homogénéité a été vérifiée par les méthodes chimiques et radiographiques et ce matériau a ensuite été embouteillé en contenants de 200 g.

Vingt-cinq laboratoires out fourni des résultats pour un ou plusieurs des 21 éléments dans chacune des deux bouteilles de CPB-1 selon un programme analytique de "libre choix". L'analyse statistique des données a donné lieu à des valeurs recommandées pour l'Ag, Al_2O_3, As, Bi, Cd, Cu, Fe, Hg, Pb, S, Sb, SiO_2 et Zn. Aussi des valeurs non-homologuées ont été déterminées pour l'Au, CaO, In, MgO, Mn, Se, Sn et Te.

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Note: Avec la collaboration de d'autres membres du personnel des Laboratoires des sciences minérales ainsi que le personnel de laboratoire d'autres organismes.

INTRODUCTION

This report describes the preparation, characterization and certification of a lead concentrate, CPB-1, for use as certified reference material. The work is one facet of the Canadian Certified Reference Materials Project (CCRMP) to certify materials that have potential value in conventional analytical or earth science laboratories. Certified reference materials issued previously by CCRMP are described in a catalogue available from CANMET, Energy, Mines and Resources Canada, Ottawa (1).

CPB-1 was chosen as a reference material because of its mineralogical complexity, and its relatively large number of minor and trace elements at useful levels of concentration. It was donated to CCRMP in late 1975 by Cominco Ltd. and was a flotation concentrate of ore from the Sullivan Mine at Kimberley, British Columbia. Its approximate mineralogical composition and particle size analysis are given in Tables 1 and 2 respectively. At the request of CCRMP, this material was analyzed for one or more of 21 elements by 25 laboratories which used methods of their choice. Recommended values for the 13 certified elements are given in Table 3; methodological, statistical and other analytical information is presented in Tables 4 to 8.

PREPARATION OF CPB-1

In early 1976, CPB-1, which had been dried at ∿100°C, was ground to pass a 74-μm screen. The powdered concentrate, weighing approximately 250 kg, was tumbled in a 570-L conical blender for approximately nine hours. Upon opening the blender, the bulk material was sampled systematically and analyzed by X-ray fluorescence and chemical methods. It was found sufficiently homogeneous to qualify for the interlaboratory program and was bottled in 200-g units. In early 1978, the bottles in storage at CANMET were each sealed under nitrogen in laminated foil pouches to provide long-term protection against oxidation.

TABLE 1

Approximate mineralogical composition of CPB-1*

Mineral	wt %
Galena	72.5
Pyrrhotite	12
Sphalerite	7
Pyrite	3
Iron Oxides	1
Aluminosilicates	1
Carbonates	1
Chalcopyrite	0.5
Boulangerite	0.5

*From J.F. Harris, Exploration Research Laboratory, Cominco Ltd., Vancouver, B.C.

TABLE 2

Particle size analysis of CPB-1 (wet screen)

Size of fraction (μm)	wt %
-74 + 55	0.6
-55 + 46	3.2
-45 + 37	1.4
-37	94.8

INTERLABORATORY PROGRAM FOR CERTIFICATION OF CPB-1

The laboratories that participated in the certification program for CPB-1 are listed alphabetically in Appendix A. Each was arbitrarily assigned a code number so that analytical results could be recorded while preserving anonymity (Table 8). The numbers bear no relation to the alphabetical order of the laboratory names.

Each laboratory was requested to submit five replicate results for each element in each of two bottles by a method of their choice and to report results on subsamples that had been dried for two hours at 105°C. Although results reported in Table 8 are on a dry basis, some laboratories deviated from the request for 10 results for each constituent. Where a laboratory submitted results for a constituent determined by more than one method, each set was considered statistically independent.

In keeping with mining industry practice, most laboratories reported aluminum, calcium, magnesium and silicon as oxides, and this form is retained in this report. When required, results for the four elements were converted to oxide equivalents. In a few cases results were not reported on a dry basis and these were subsequently corrected for the 0.14% moisture content of CPB-1.

It was arbitrarily decided not to assign a recommended value for those constituents for which fewer than 10 sets of results were submitted. This accounts for the different treatments accorded the constituents listed in Tables 3 and 4.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

Detection of outliers

Sets of results whose means differed by more than twice the overall standard deviation from the initial mean value for that constituent were not used for subsequent computations to avoid possible biasing of the statistics. Sets with unusually high variance were examined for individual outlying results and such results were deleted if they caused the mean of the set to be further from the overall mean. In extreme cases, entire sets with high variance were rejected. Other sets were rejected for methodological reasons and are discussed below. All results that were not used are identified in Table 8.

Confirmation of homogeneity using interlaboratory results

Table 7 gives the means and coefficients of variation for each set of results for constituents assigned recommended values. Also given are the results of the *t*-tests of differences between bottles at the 5% significance level. Rejection of the null hypothesis of no difference between bottle means is signified by the code REJECT. For the 13 constituents certified, the rejection rate was 15%. This is somewhat higher than is usually encountered in CCRMP certification programs. However the between-bottle components of variance for the certified elements, estimated by a two-way analysis of variance, are small and considered unimportant for most applications of CPB-1 (Table 7). For certain critical applications, it may be necessary to take the between bottles variation into account. The degree of homogeneity of CPB-1 is also illustrated in Fig. 1, in which, for each set, the difference between the means of the results for the two bottles is plotted against the corresponding mean of the results for both bottles. The vertical bar represents the 95% confidence interval of the former. If a bar does not intersect the abscissa, the null hypothesis is rejected.

Estimation of consensus values and 95% confidence limits

A one-way analysis of variance technique was used to calculate the consensus values (means) and their variance. The analytical data were assumed to fit the following model (2):

$$x_{ij} = \mu + y_i + e_{ij}$$

where:

x_{ij} = the j^{th} result reported in set i;

μ = the true consensus value that is estimated by the overall mean $\bar{x}..$;

y_i = the discrepancy between the mean of the results from set i ($\bar{x}_i.$) and μ; and

e_{ij} = the discrepancy between x_{ij} and $\bar{x}_i.$

It is assumed in this analysis that both y_i and e_{ij} are normally distributed with means of zero and variances of ω^2 and σ^2, respectively. The significance of ω^2 can be detected by comparing the ratio of between-set mean squares to within-set mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom. The magnitude of ω^2 and σ^2 can be estimated from the ANOVA table.

The consensus value in the above model can be estimated by the overall mean $\bar{x}..$, thus:

$$\bar{x}.. = \frac{\sum_i^k \sum_j^{n_i} x_{ij}}{\sum_i^k n_i}$$

with the variance of the overall mean being given by:

$$V\,[\bar{x}..] = \frac{\sum_i^k n_i^{\,2}}{\left(\sum_i^k n_i\right)^2}\,\omega^2 + \frac{\sigma^2}{\sum_i^k n_i}$$

The 95% confidence limits for the overall mean are then given by:

$$\bar{x}.. \pm \left[t_{0.975,(k-1)} \cdot \sqrt{[V\ \bar{x}..]}\right]$$

where:

n_i = the number of results reported in set i;

k = the number of sets.

Analysis of variance and expected mean squares for the one-way classification

Source of variance	Sums of squares	Degrees of freedom	Mean squares	E [Mean squares]
Between-sets	$\sum_{i}^{k} n_i (\bar{x}_{i.} - \bar{x}_{..})^2$	k-1	$s_2^{\,2}$	$\sigma^2 + \frac{1}{k-1}\left(\sum_{i}^{k} n_i - \frac{\sum_{i}^{k} n_i^{\,2}}{\sum_{i}^{k} n_i}\right)\omega^2$
Within-sets	$\sum_{i}^{k}\sum_{j}^{n_i} (x_{ij} - \bar{x}_{i.})^2$	$\sum_{i}^{k} n_i - k$	$s_1^{\,2}$	σ^2
Total	$\sum_{i}^{k}\sum_{j}^{n_i} (x_{ij} - \bar{x}_{..})^2$	$\sum_{i}^{k} n_i - 1$		

The above values and other statistics computed from the one-way ANOVA are presented in Tables 3 and 4.

Certification factor

The certification factor (CF) is a measure for evaluating the quality of reference materials issued by CCRMP (3). It is computed from the following expression:

$$CF = 200 \left[t_{0.975,(k-1)} \cdot \sqrt{V\,[\bar{x}_{..}]} \right] / \bar{x}_{..} / \overline{cv}$$

where $\overline{cv}$ is the average of the within-set coefficients of variation and is given by:

$$\overline{cv} = \sum_{i}^{k} cv_i / k$$

The critical value of CF is 4. If a selected constituent has a CF greater than 4, the reference material is considered unacceptable with respect to that constituent.

The CF for the 13 certified constituents of CPB-1 are given in Table 3 along with the consensus values which are boxed in for easy identification.

Similar statistics for eight non-certified constituents are given in Table 4.

Discussion of analytical results

An outline of the principal titrimetric methods used for lead in CPB-1 is given in Appendix B.

Table 5 gives a methodological classification of results (outliers excluded) where there is a clear-cut distinction between types of method, particularly in decomposition, separations and determinative steps. In some cases however, a single method, with minor variations was used for one or more elements by all participants. The differences in the sub-sample decomposition and in the conditioning of solutions do not warrant a detailed listing in Table 5. However, some general comments on the determination of these elements are given below.

Alumina, lime and magnesia

Multi-element analysis of a single subsample solution was used by most participants for determining Al_2O_3, CaO and MgO. In some cases the solution was also used for determining other elements. Approximately one half of the participants used a mixed-acid decomposition involving hydrofluoric acid to decompose siliceous gangue constituents. The other half used an alkali fusion after a preliminary acid treatment of the subsample. Similarly, about half the results were obtained on solutions to which La or Sr had been added as a buffer or releasing agent. The variations in decomposition or conditioning of the sample solution did not lead to significant differences in results.

It should be noted that several sets of results for each of Al_2O_3, CaO and MgO were rejected prior to computations because it was either known or suspected that they were obtained by methods that did not involve a hydrofluoric acid treatment or an alkaline fusion of gangue minerals. In most of these cases the deleted results were lower than the corresponding consensus values.

Although the concentrations of Al_2O_3, CaO and MgO are similar, Tables 3 and 4 show that only Al_2O_3 has a certification factor less than the acceptable value of 4. The factors for CaO and MgO exceed this limit mainly because of the large spread in the results. It is noteworthy, however, that, based on the experience of a large number of previous CCRMP interlaboratory studies, the $\overline{cv}$ for both Al_2O_3 and CaO are higher than expected for elements at their level of concentration. For Al_2O_3 it is the large $\overline{cv}$ together with the high spread that gives an acceptable certification factor.

Copper and cadmium

With few exceptions Cu and Cd were determined by atomic absorption spectrophotometry on the same subsample solution which, in some cases, was also used for other determinations. Nearly all participants used an oxidizing acid mixture for decomposition. Results were not dependent on the use of hydrofluoric acid to decompose gangue minerals, and this agrees with the two elements occurring in free particles of sulphide minerals.

Mercury

The cold-vapour atomic absorption method was used by 12 of 14 laboratories to obtain results for mercury (4). Most analysts used a low-temperature decomposition involving a mixture of nitric and hydrochloric acids; some also used a second oxidizing agent such as $KMnO_4$, $KClO_3$ or Br_2. Stannous chloride was the most common reagent for reducing mercury in the cold-vapour generator. It should be noted that, with the exception of Lab 5, all participants analyzed subsamples that had been previously dried at 105°C for 2 h.

Sulphur

Thirteen of the 16 sets of results for sulphur were obtained by the classical gravimetric method involving the weighing of $BaSO_4$. Ten laboratories used acid decomposition involving nitric acid and bromine, and three used peroxide or carbonate fusion. Lead was separated as the carbonate by most analysts. No relationship is evident between the results and the variations in decomposition or in the treatment of the sample solution prior to the precipitation of $BaSO_4$.

Stability of CPB-1

Figure 2 shows the laboratory means for lead in CPB-1 plotted against the date on which the analyses were reported. Clearly, there is no trend over the 21-month reporting period that suggests decreasing values due to oxidation of the sulphides (mainly to sulphates) comprising CPB-1. It is known, however, that unprotected samples of many sulphide ores and concentrates are susceptible to oxidation under ambient conditions of use and storage in a laboratory atmosphere (5). To protect the stock of CPB-1 stored at CANMET, all bottles were sealed under nitrogen in individual laminated foil pouches in April 1978. This procedure should ensure indefinitely the validity of the recommended values for CPB-1 given in Table 3. This conclusion is supported by the data plotted in Fig. 3 which show that there was no change in weight of protected test bottles from June 1, 1978 to March 15, 1979. Monitoring will be continued throughout the life of the stock of CPB-1.

Figure 3 also shows that unprotected test bottles of CPB-1 deliberately opened and exposed to the atmosphere in the storage room at CANMET, gained approximately 0.2% in weight during the above monitoring period. It is strongly recommended, therefore, that users store opened bottles of CPB-1 under a dry inert gas in a desiccator jar or in a new heat-sealed foil pouch. Also, when taking subsamples, the contents of the bottle should be exposed to air for the shortest time possible.

REFERENCES

1. Faye, G.H. "Certified and provisional reference materials available from the Canada Centre for Mineral and Energy Technology, 1978"; CANMET Report 78-3; CANMET, Energy, Mines and Resources Canada; 1978.

2. Brownlee, K.A. "Statistical theory and methodology in science and engineering"; John Wiley and Sons, Inc. New York; 1960.

3. Sutarno, R. and Faye, G.H. "A measure for assessing certified reference ores and related materials"; Talanta; 22:676-681; 1975.

4. Hatch, W.R. and Ott, W.L. "Determination of sub-microgram quantities of mercury by atomic absorption spectrophotometry"; Anal Chem; 40:2085-2087; 1968.

5. Faye, G.H. and Steger, H.F. "A case study of the ambient oxidation of two zinc-lead (sulphide) reference ores"; Talanta; In press; 1979.

TABLE 3

Recommended values and associated statistical parameters
(outliers excluded)

Element (oxide)	N	n	$\bar{x}$	95% CL low	95% CL high	Spread %	$\overline{cv}$ %	CF
			(wt %)	(wt %)	(wt %)			
Al_2O_3	10	108	0.28	0.27	0.30	10.4	3.2	3.3
As	17	167	0.056	0.052	0.059	13.7	6.0	2.3
Bi	15	151	0.023	0.021	0.024	10.5	3.8	2.8
Cd	13	140	0.0143	0.0138	0.0148	7.0	2.6	2.7
Cu	22	231	0.254	0.250	0.258	3.0	1.5	2.1
Fe	19	188	8.43	8.37	8.49	1.5	0.4	3.4
Pb	27	278	64.74	64.62	64.86	0.4	0.2	2.0
S	15	136	17.8	17.7	18.0	1.3	0.5	2.7
Sb	13	119	0.36	0.34	0.37	7.8	2.2	3.5
SiO_2	13	128	0.74	0.70	0.77	9.1	3.8	2.4
Zn	27	249	4.42	4.38	4.46	1.8	1.0	1.7
			(µg/g)	(µg/g)	(µg/g)			
Ag	22	203	626	620	632	1.9	1.0	2.0
Hg	14	127	5.5	5.0	5.9	17.6	9.2	1.9

N = number of sets; n = number of results; $\bar{x}$ = overall mean (recommended value); CL = confidence limits; Spread = 95% confidence interval as percentage of mean; $\overline{cv}$ = average within-lab coefficient of variation; CF = certification factor (see page 3).

TABLE 4

Means and associated statistical parameters for constituents not certified (outliers excluded)

Element (oxide)	N	n	$\bar{x}$	95% CL low	95% CL high	Spread %	$\overline{cv}$ %	CF
			(wt %)	(wt %)	(wt %)			
CaO	9	99	0.88	0.84	0.91	9	2	4
MgO	11	118	0.15	0.145	0.164	12	2	6
Mn	13	142	0.039	0.037	0.041	11	3	4
Sn	6	59	0.022	0.016	0.029	59	6	10
			(µg/g)	(µg/g)	(µg/g)			
Au	4	53	0.17	0	0.35	200	7	29
In	6	53	14	11	17	42	12	4*
Se	7	66	31	28	34	21	4	5
Te	3	24	0.7	0.0	1.7	310	6	54

N = number of sets; n = number of results; $\bar{x}$ = overall mean; Spread = 95% confidence interval as percentage of mean; $\overline{cv}$ = average within-lab coefficient of variation; CF = certification factor (see page 3).

*CF actually slightly less than 4; however, In not certified because of insufficient data.

TABLE 5(a)

Summary of analytical methods for Ag in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (µg/g)
Fire assay	Classical fire assay with gravimetric finish	7	5,6,14,18,21, 34,37	68	627
Fire assay-atomic abs.					
	Pb button scorified to ∿ 2 g; dissolved in HNO_3 for a.a.	1	1	10	620
	Pb button cupelled to bead; dissolved in HNO_3 for a.a.	1	9	6	599
	Sample leached in HNO_3; soln analyzed by a.a.; residue assayed to give Pb button; dissolved in HNO_3 for a.a.	1	12	10	641
	Pb button partially cupelled; dissolved in HNO_3 for a.a.; Ag loss det'd with Ag-110	1	12	10	633
	Pb button partially cupelled; dissolved in HNO_3 for a.a.	1	16	10	602
Atomic absorption					
	HNO_3 + HCl (some with Br_2); final soln 10-40% v/v HCl	5	24,30,31,33, 40	50	625
	HNO_3 (one with HF); final soln dilute HNO_3	2	26,38	20	621
	Details not given	1	19	10	643
	HNO_3 + Br_2; Ag complexed with Hg	1	35	4	636
Emission spectrographic		1	39	5	635

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(b)

Summary of analytical methods for As in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	$HNO_3 + HCl + H_2SO_4$; final soln 4% v/v HCl; generation of AsH_3	1	38	10	0.060
	$HNO_3 + Br_2$; final soln dilute HNO_3; graphite furnace	1	16	10	0.050
	$HNO_3 + HCl$; final soln dilute $HNO_3 + HCl$	1	30	10	0.065
Colorimetric (spectrophotometric)					
Arseno-molybdate	$HNO_3 + Br_2 + HCl + H_2SO_4$; As coppt'd with Fe, ext'd as xanthate	1	3	10	0.061
	$HNO_3 + HCl + KClO_3$; As sep'd by distillation	1	23	22	0.058
	$HNO_3 + H_2SO_4$; As sep'd by distillation	2	18,34	18	0.051
	Na_2O_2 fusion; As ext'd with benzene from 9M HCl	1	24	10	0.059
Dithiocarbamate	$KHSO_4$ fusion; As sep'd by distillation	1	9	10	0.062
	Na_2O_2 fusion; As sep'd by distillation	1	5	10	0.042
Iodide	$HNO_3 + HClO_4$; As ext'd with cyclohexane from HCl-iodide soln	1	40	10	0.058
Titrimetric					
Iodometric	$HNO_3 + H_2SO_4$; As sep'd by distillation	2	33,35	14	0.054
	$HNO_3 + HCl + H_2SO_4$; As coppt'd with Fe, sep'd by distillation	1	21	10	0.050
Emission spectrographic		1	39	5	0.065
Neutron activation		1	24	10	0.049
Not specified		1	14	10	0.059

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(c)

Summary of analytical methods for Bi in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	HNO_3 + HCl and/or $HClO_4$ + HF; final soln dilute HCl, HNO_3 or $HClO_4$	3	5,19,34	30	0.021
	HNO_3 + HCl and/or $HClO_4$; final soln dilute HCl or HNO_3	5	6,14,21,30,35	48	0.023
	HNO_3 + HCl; Bi sep'd as sulphide; final soln dilute HCl	1	23	20	0.024
	$HClO_4$ + HF; insol fused with Na_2CO_3; solns combined, dilute HCl	1	34	10	0.021
Colorimetric (spectrophotometric)					
Iodide	Na_2O_2 fusion; Bi ext'd as dithiocarbamate	1	3	10	0.022
Bromide	HNO_3 + Br_2 + H_2SO_4; Bi ext'd with tri-n-octylamine (benzene)	1	24	10	0.027
Emission spectrographic					
		2	39,40	15	0.023
	Fire assay collection of Bi in Pb	1	18	8	0.019

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(d)

Summary of methods for Fe in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Titrimetric					
Dichromate	HNO_3 + HCl + H_2SO_4; Fe ppt'd with NH_3, reduced with $SnCl_2$	4	24,29,31,34	40	8.48
	HNO_3 + HCl + HF + H_2SO_4 (or $HClO_4$); Fe ppt'd with NH_3, reduced with $SnCl_2$	2	1,21	20	8.39
	Na_2O_2 fusion; cake dissolved in dilute HCl; Fe ppt'd with NH_3, reduced with $SnCl_2$	3	5,6,23	30	8.47
	Na_2O_2 fusion; other details not given	1	35	4	8.33
	Decomposition not given, Fe reduced with H_2S	1	18	12	8.52
	Details not given	2	13,14	20	8.44
Permanganate	Sample roasted, treated with HNO_3 + HCl + HF + H_2SO_4; Fe reduced with $SnCl_2$	1	26	10	8.59
Ceric amm. sulphate	Na_2O_2 + NaOH fusion; cake dissolved in dilute HCl; Fe ppt'd with NH_3, Fe reduced with Pb°	1	9	10	8.59
Atomic absorption					
	HNO_3 + HCl + HF + $HClO_4$; Fe + Pb ppt'd with NH_3-$(NH_4)_2CO_3$, dissolved in HNO_3; insol. fused with Na_2O_2 combined with above	1	1	10	8.33
	HNO_3 + HCl + HF; final soln 1% v/v HNO_3 containing EDTA	1	40	12	8.21
	HNO_3 (fuming); dilution with H_2O	1	38	10	8.32
	Na_2O_2 fusion; cake dissolved in dilute HCl	1	2	10	8.24

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(e)

Summary of analytical methods for Pb in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Titrimetric					
EDTA	HNO_3 + HCl + H_2SO_4; Pb isolated as $PbSO_4$, dissolved in acetate or chloride medium and titrated either at room temp or near boiling point (see Appendix B)	5	1,1a,5,18, 37	52	64.83
	HBr + Br; as above	1	35	12	64.79
	HNO_3 + HCl + HF + H_2SO_4; Pb isolated by extn as diethyldithiocarbamate	1	45	10	64.65
	HNO_3 + HCl + HF + Br_2; no other details given	1	32	10	64.59
	HNO_3 + HCl + H_2SO_4; Pb isolated as $PbSO_4$, after dissolution in acetate titrated potentiometrically	1	16	10	64.63
Molybdate	HNO_3 + HCl + H_2SO_4 (two also with HF); Pb isolated as $PbSO_4$, dissolved in acetate and titrated using tannic acid as external indicator (see Appendix B)	7	6,9,13,21, 23,34,40	79	64.81
	HNO_3 + $KClO_4$ + HBr; Pb isolated as PbS which, after dissolution, titrated as above	1	30	10	64.91
	Details not given	1	14	10	65.17
Chromate-Iodometric	HNO_3 + HCl + HF + H_2SO_4; Pb isolated as $PbSO_4$, converted to $PbCrO_4$ and dissolved in HCl medium containing iodide; detn completed by titration with thiosulphate (see Appendix B)	1	26	10	64.09
Atomic absorption					
	Na_2O_2 fusion; cake dissolved in HCl; a.a. by slanted burner technique	1	2	10	64.92
	HNO_3 + HCl + $HClO_4$; no other details given	2	19,26	20	64.66
Gravimetric					
	HNO_3 + HCl + H_2SO_4; Pb isolated as $PbSO_4$, dissolved in amm. acetate; ppt'd as $PbCrO_4$ and weighed	3	24,29,39	25	64.70
	HNO_3 + Br_2 + HCl + H_2SO_4; Pb leached from weighed $PbSO_4$ + insol, det'd by difference	1	33	10	64.53
	HNO_3 + HCl; Pb ppt'd as $PbSO_4$, no other details given	1	38	10	64.35

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(f)

Summary of analytical methods for Sb in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	HNO_3; final soln 2% w/v tartaric + 12% v/v HCl	1	9	10	0.33
	HNO_3; final soln dilute tartaric	1	21	10	0.35
	HCl + $KClO_3$; final soln dilute tartaric + HCl	1	6	10	0.40
	HNO_3 + HCl + $HClO_4$; final soln 10% v/v HCl	1	14	10	0.37
	HNO_3 + HF; final soln 10% v/v HNO_3	1	5	10	0.37
	HNO_3 + HCl; no other details given	2	30,35	14	0.37
	HNO_3 + H_2SO_4; Sb sep'd by distillation; final soln dilute HCl + HBr	1	34	10	0.33
	Na_2O_2 fusion; final soln dilute HCl	1	24	10	0.37
Colorimetric (spectrophotometric)					
Iodide	HNO_3 + HCl + H_2SO_4; Sb coppt'd with Fe, ext'd as xanthate	1	3	10	0.37
Rhodamine B	Na_2O_2 fusion; Sb ext'd as xanthate	1	23	10	0.35
Emission spectroscopic		2	39,40	15	0.33

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(g)

Summary of analytical methods for SiO_2 in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Gravimetric					
	HCl (+ HNO_3) + $HClO_4$; residue fused with Na_2O_2 or Na_2CO_3; cake dissolved in HCl; SiO_2 dehydrated with $HClO_4$; volatilized with HF	3	1,4,34	30	0.73
	HNO_3 + HCl (or HBr) + H_2SO_4; insol + $PbSO_4$ treated with acetate, volatilized with HF	3	24,29,35	28	0.70
	Leach with HCl or dilute HNO_3; residue fused with Na_2O_2 or Na_2CO_3; cake dissolved in HCl; SiO_2 dehydrated with $HClO_4$ or HCl; volatilized with HF	2	5,19	20	0.70
	Na_2O_2 fusion, cake dissolved in HCl; SiO_2 dehydrated with $HClO_4$ and weighed directly; no HF	2	14,23	20	0.82
	Na_2O_2 fusion; no other details given	1	13	10	0.78
Atomic absorption					
	Na_2O_2 sinter; cake dissolved in 5% v/v HCl	1	2	10	0.73
	Li metaborate fusion; cake dissolved in HF + H_3BO_3	1	26	10	0.76

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 5(h)

Summary of analytical methods for Zn in CPB-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Titrimetric					
EDTA	HNO_3 + HCl + one or more of: Br_2, HF, HCl, $HClO_4$; zinc sep'd by extn with MIBK	4	4,18,24,33	35	4.56
	HNO_3 + HCl + H_2SO_4; Fe removed by pptn with NH_3 or NaOH	2	5,39	14	4.33
	HNO_3 + Br_2 + HCl + H_2SO_4; Zn sep'd by anion exchange	1	45	10	4.42
Ferrocyanide	HNO_3 + Br_2 + H_2SO_4; Fe removed with NH_3; Zn sep'd as sulphide	1	33	6	4.36
	HNO_3 + HCl + $KClO_3$; Fe removed with NH_3; Cu removed with Pb°	1	30	10	4.41
	Details not given	2	13,14	20	4.50
Ferrocyanide-amperometric	HNO_3 + Br_2 + HCl + H_2SO_4; Fe removed with NH_3	1	45	10	4.45
Atomic absorption					
	HNO_3 + HCl and/or HF + $HClO_4$; final soln dilute $HClO_4$	7	1,6,9,16,19, 21,34	69	4.39
	HNO_3 + HCl + HF; final soln dilute HNO_3	2	26,40	22	4.37
	HNO_3 + Br_2 + HCl; final soln dilute HNO_3	1	31	10	4.34
	Na_2O_2 fusion; cake dissolved in dilute HCl or HNO_3	2	2,24	20	4.51
	HNO_3 (fuming)	1	38	10	4.31
	HNO_3 + HCl + H_2SO_4	1	39	5	4.26
	HBr + Br_2; Pb sep'd as $PbSO_4$	1	35	8	4.37

N = number of laboratories; n = number of results; $\bar{x}$ = overall mean.

TABLE 6

Methodological classification for elements not certified (outliers excluded)

Element (oxide)	Method	N	n	$\bar{x}$	Spread %	$\overline{cv}$, %
				(wt %)		
CaO						
	AA	9	99	0.88	8.7	2.1
MgO						
	AA	11	118	0.15	12.0	2.0
Mn						
	AA	12	132	0.039	11.5	2.6
	COLOR	1	10	0.040	--	0.9
Sn						
	AA	2	30	0.02	--	8
	COLOR	1	10	0.02	--	7
	ES	3	19	0.03	112	4
				(μg/g)		
Au						
	FA-AA	3	43	0.2	310	9
	FA	1	10	0.1	--	--
In						
	AA	3	28	14	110	11
	COLOR	1	10	12	--	13
	ES	2	15	13	--	13
Se						
	AA	3	26	31	34	5
	COLOR	4	40	31	45	3
Te						
	AA	1	10	0.5	--	12
	COLOR	2	14	0.8	--	3

AA = atomic absorption; FA-AA = fire assay-atomic absorption; COLOR = colorimetric (spectrophotometric); ES = emission spectrographic.

N = number of sets; n = number of results; $\bar{x}$ = overall mean of sets; Spread = 95% confidence interval as percentage of mean; $\overline{cv}$ = average within-lab coefficient of variation.

TABLE 7

Laboratory means, coefficients of variation and summary of t-test on between bottle results for certified constituents

Ag (μg/g)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(FA-AA)	5	611.8000	8.6718	5	628.2000	3.8341	REJECT	10	620.0000	10.7083	1.73
LAB- 5	(FA)	5	610.3000	2.8627	5	609.4800	2.9474	A	10	609.8900	2.7731	.45
LAB- 6	(FA)	5	627.4000	2.8810	5	621.2000	5.8907	A	10	624.3000	5.4579	.87
LAB- 9	(FA-AA)	3	602.3000	3.7403	3	595.9000	1.4000	A	6	599.1000	4.3206	.72
LAB-12	(FA-AA)	5	642.4000	2.8810	5	640.4000	1.8166	A	10	641.4000	2.5033	.39
LAB-12	(FA-AA)	5	632.8000	4.2071	5	634.0000	7.3144	A	10	633.4000	5.6608	.89
LAB-14	(FA)	5	624.0000	6.1644	5	620.2000	3.0332	A	10	622.1000	4.9989	.80
LAB-16	(FA-AA)	5	603.0000	2.4495	5	600.0000	7.0356	A	10	601.5000	5.2122	.87
LAB-18	(FA)	4	645.0000	10.0000	4	650.0000	11.5470	A	8	647.5000	10.3510	1.60
LAB-19	(AA)	5	641.8000	6.2209	5	643.8000	1.7889	A	10	642.8000	4.4422	.69
LAB-21	(FA)	5	637.4000	3.5777	5	626.4000	2.5100	REJECT	10	631.9000	6.4885	1.03
LAB-24	(AA)	5	614.2000	1.0954	5	613.6000	3.7815	A	10	613.9000	2.6437	.43
LAB-26	(AA)	5	628.8000	4.1473	5	628.4000	4.3359	A	10	628.6000	4.0056	.64
LAB-30	(AA)	5	611.2000	9.0111	5	620.8000	3.9623	A	10	616.0000	8.2865	1.35
LAB-31	(AA)	5	618.0000	8.3666	5	622.0000	4.4721	A	10	620.0000	6.6667	1.08
LAB-33	(AA)	5	626.6000	2.7928	5	626.6000	2.5100	A	10	626.6000	2.5033	.40
LAB-34	(FA)	5	627.9000	5.5915	5	629.9200	6.0586	A	10	628.9100	5.5985	.89
LAB-35	(AA)	2	635.0000	1.4142	2	637.5000	.7071	A	4	636.2500	1.7078	.27
LAB-37	(FA)	5	630.1200	2.7298	5	629.4000	2.2045	A	10	629:7600	2.3698	.38
LAB-38	(AA)	5	620.0000	10.0000	5	606.0000	5.4772	REJECT	10	613.0000	10.5935	1.73
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	635.0000	15.8114	2.49
LAB-40	(AA)	5	648.0000	16.8077	5	652.0000	5.7009	A	10	650.0000	12.0185	1.85

Variance between sets, between bottles and within bottles = 1.40×10^{2}, 1.53×10^{1} and 3.26×10^{1}, respectively.

Al_2O_3 (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(AA)	5	.3400	.0141	5	.3160	.0219	A	10	.3280	.0215	6.55
LAB- 4	(AA)	5	.2868	.0053	5	.2866	.0030	A	10	.2867	.0041	1.41
LAB- 5	(AA)	5	.2800	.0000	5	.2780	.0045	A	10	.2790	.0032	1.13
LAB- 6	(AA)	5	.2840	.0055	5	.2940	.0055	REJECT	10	.2890	.0074	2.55
LAB- 9	(AA)	5	.2960	.0089	5	.2720	.0110	REJECT	10	.2840	.0158	5.56
LAB-14	(AA)	5	.2812	.0018	5	.2802	.0023	A	10	.2807	.0020	.71
LAB-18	(AA)	4	.3050	.0129	4	.3025	.0126	A	8	.3038	.0119	3.91
LAB-19	(AA)	5	.1880	.0045	5	.1960	.0055	REJECT	10	.1920	.0063	3.29
LAB-21	(AA)	5	.3520	.0084	5	.3420	.0148	A	10	.3470	.0125	3.61
LAB-26	(AA)	5	.2660	.0134	5	.2700	.0141	A	10	.2680	.0132	4.91
LAB-23	(AA)	10	.2580	.0079	10	.2610	.0088	A	20	.2595	.0083	3.18
LAB-34	(AA)	5	.2850	.0068	5	.2892	.0022	A	10	.2871	.0053	1.83
LAB-35	(AA)	2	.1750	.0071	2	.1700	.0141	A	4	.1725	.0096	5.55

Variance between sets, between bottles and within bottles = 2.96×10^{-4}, 5.51×10^{-5} and 8.31×10^{-5}, respectively.

As (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 3	(COLOR)	5	.0613	.0004	5	.0615	.0004	A	10	.0614	.0003	.57
LAB- 5	(COLOR)	5	.0440	.0055	5	.0400	0.0000	A	10	.0420	.0042	10.04
LAB- 6	(COLOR)	5	.0883	.0022	5	.0884	.0046	A	10	.0883	.0034	3.87
LAB- 9	(COLOR)	5	.0604	.0057	5	.0638	.0037	A	10	.0621	.0049	7.83
LAB-13	(TITR)	5	.1815	.0018	5	.1823	.0025	A	10	.1819	.0021	1.16
LAB-14		3	.0593	.0006	3	.0593	.0006	A	6	.0593	.0005	.87
LAB-16	(AA)	5	.0497	.0012	5	.0499	.0010	A	10	.0498	.0011	2.13
LAB-18	(COLOR)	4	.0585	.0031	4	.0583	.0021	A	8	.0584	.0024	4.19
LAB-21	(TITR)	5	.0482	.0047	5	.0508	.0019	A	10	.0495	.0036	7.33
LAB-23	(COLOR)	11	.0622	.0046	11	.0533	.0105	REJECT	22	.0577	.0092	15.87
LAB-24	(COLOR)	5	.0585	.0047	5	.0587	.0067	A	10	.0586	.0055	9.31
LAB-24	(NAA)	6	.0491	.0021	6	.0481	.0028	A	12	.0486	.0025	5.05
LAB-30	(AA)	5	.0650	.0014	5	.0658	.0004	A	10	.0654	.0011	1.64
LAB-33	(TITR)	5	.0556	.0044	5	.0596	.0026	A	10	.0576	.0040	7.00
LAB-34	(COLOR)	5	.0466	.0029	5	.0429	.0019	REJECT	10	.0448	.0030	6.77
LAB-35	(TITR)	2	.0500	0.0000	2	.0400	0.0000	***R**	4	.0450	.0058	12.83
LAB-38	(AA)	5	.0586	.0019	5	.0606	.0019	A	10	.0596	.0021	3.48
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.0650	.0035	5.44
LAB-40	(COLOR)	5	.0582	.0013	5	.0586	.0016	A	10	.0584	.0014	2.38

Variance between sets, between bottles and within bottles = 5.13×10^{-5}, 1.00×10^{-6} and 1.72×10^{-5}, respectively.

TABLE 7 (cont'd)

Bi (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(AA)	5	.0283	.0015	5	.0295	.0014	A	10	.0289	.0015	5.34
LAB- 3	(COLOR)	5	.0217	.0003	5	.0213	.0003	A	10	.0215	.0004	1.64
LAB- 5	(AA)	5	.0200	0.0000	5	.0200	0.0000	***R**	10	.0200	.0000	.00
LAB- 6	(AA)	5	.0206	.0005	5	.0213	.0003	REJECT	10	.0209	.0005	2.58
LAB- 9	(AA)	5	.0340	.0007	5	.0314	.0017	REJECT	10	.0327	.0018	5.59
LAB-13	(AA)	5	.3245	.0135	5	.3245	.0135	A	10	.3245	.0127	3.91
LAB-14	(AA)	3	.0197	.0008	3	.0184	.0018	A	6	.0191	.0014	7.49
LAB-18	(ES)	4	.0198	.0013	4	.0185	.0010	A	8	.0191	.0012	6.52
LAB-19	(AA)	5	.0217	.0003	5	.0219	.0002	A	10	.0218	.0003	1.18
LAB-21	(AA)	5	.0250	.0000	5	.0250	.0000	A	10	.0250	.0000	.00
LAB-23	(AA)	10	.0235	.0005	10	.0239	.0006	A	20	.0237	.0006	2.41
LAB-24	(COLOR)	5	.0267	.0010	5	.0264	.0018	A	10	.0266	.0014	5.24
LAB-30	(AA)	5	.0237	.0002	5	.0235	.0002	A	10	.0236	.0002	.87
LAB-34	(AA)	5	.0213	.0002	5	.0211	.0001	REJECT	10	.0212	.0002	.92
LAB-35	(AA)	6	.0233	.0013	6	.0225	.0020	A	12	.0229	.0017	7.22
LAB-38	(AA)	5	.0245	.0007	5	.0242	.0007	A	10	.0243	.0007	2.83
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.0230	.0025	10.76
LAB-40	(ES)	5	.0224	.0018	5	.0224	.0017	A	10	.0224	.0016	7.35

Variance between sets, between bottles and within bottles = 3.57×10^{-6}, 0 and 8.14×10^{-7}, respectively.

Cd (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(AA)	5	.0150	.0000	5	.0150	.0000	A	10	.0150	.0000	.00
LAB- 5	(AA)	5	.0152	.0004	5	.0152	.0004	A	10	.0152	.0004	2.77
LAB- 6	(AA)	5	.0143	.0001	5	.0143	0.0000	A	10	.0143	.0000	.30
LAB- 9	(AA)	5	.0134	.0005	5	.0148	.0004	REJECT	10	.0141	.0009	6.21
LAB-13	(AA)	5	.0100	0.0000	5	.0100	0.0000	***R**	10	.0100	.0000	.00
LAB-14	(AA)	5	.0146	.0005	5	.0146	.0005	A	10	.0146	.0005	3.54
LAB-18	(AA)	4	.0140	0.0000	2	.0140	0.0000	***R**	6	.0140	.0000	.00
LAB-19	(AA)	5	.0147	.0002	5	.0149	.0001	A	10	.0148	.0002	1.33
LAB-21	(AA)	5	.0144	.0005	5	.0140	.0000	A	10	.0142	.0004	2.97
LAB-23	(AA)	10	.0131	.0010	10	.0130	.0011	A	20	.0131	.0010	7.65
LAB-30	(AA)	5	.0188	.0016	5	.0184	.0013	A	10	.0186	.0014	7.69
LAB-34	(AA)	5	.0145	.0001	5	.0147	.0002	REJECT	10	.0146	.0002	1.23
LAB-35	(AA)	6	.0132	.0003	6	.0135	.0003	A	12	.0134	.0003	2.40
LAB-38	(AA)	5	.0147	.0002	5	.0148	.0002	A	10	.0148	.0002	1.42
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.0159	.0022	13.81
LAB-40	(AA)	6	.0156	.0006	6	.0156	.0005	A	12	.0156	.0005	3.30

Variance between sets, between bottles and within bottles = 8.24×10^{-7}, 7.58×10^{-8} and 1.21×10^{-7}, respectively.

Cu (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(COLOR)	5	.2564	.0046	5	.2578	.0036	A	10	.2571	.0039	1.53
LAB- 1	(AA)	5	.2600	.0000	5	.2600	.0000	A	10	.2600	.0000	.00
LAB- 1	(AA)	5	.2620	.0045	5	.2620	.0045	A	10	.2620	.0042	1.61
LAB- 2	(AA)	5	.2504	.0023	5	.2540	.0021	REJECT	10	.2522	.0028	1.12
LAB- 3	(COLOR)	5	.2668	.0034	5	.2660	.0014	A	10	.2664	.0025	.94
LAB- 5	(AA)	5	.2400	.0000	5	.2400	.0000	A	10	.2400	.0000	.00
LAB- 6	(AA)	5	.2446	.0005	5	.2430	.0027	A	10	.2438	.0020	.84
LAB- 9	(AA)	5	.2334	.0070	5	.2492	.0066	REJECT	10	.2413	.0105	4.36
LAB-14	(AA)	5	.2442	.0023	5	.2452	.0022	A	10	.2447	.0022	.88
LAB-18	(AA)	4	.2600	.0082	4	.2625	.0050	A	8	.2613	.0064	2.45
LAB-19	(AA)	5	.2578	.0013	5	.2594	.0019	A	10	.2586	.0018	.69
LAB-21	(AA)	5	.2488	.0013	5	.2468	.0008	REJECT	10	.2478	.0015	.60
LAB-23	(AA)	10	.2600	.0000	9	.2622	.0067	A	19	.2611	.0046	1.76
LAB-24	(AA)	5	.2480	.0045	5	.2460	.0055	A	10	.2470	.0048	1.96
LAB-26	(AA)	5	.2540	.0014	5	.2540	.0021	A	10	.2540	.0017	.67
LAB-30	(AA)	5	.2526	.0025	5	.2480	.0037	A	10	.2503	.0039	1.54
LAB-31	(AA)	5	.2620	.0045	5	.2620	.0045	A	10	.2620	.0042	1.61
LAB-32	(AA)	5	.2708	.0035	5	.2678	.0018	A	10	.2693	.0031	1.14
LAB-34	(AA)	5	.2482	.0027	5	.2470	.0007	A	10	.2476	.0020	.79
LAB-35	(AA)	6	.2600	.0110	6	.2583	.0117	A	12	.2592	.0108	4.18
LAB-38	(AA)	5	.2474	.0042	5	.2422	.0034	A	10	.2448	.0045	1.86
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	.2200	.0187	8.50
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	.1320	.0045	3.39
LAB-40	(AA)	6	.2468	.0035	6	.2471	.0037	A	12	.2470	.0035	1.40

Variance between sets, between bottles and within bottles = 7.04×10^{-5}, 5.13×10^{-6} and 1.51×10^{-5}, respectively.

TABLE 7 (cont'd)

Fe (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(TITR)	5	8.3760	.0251	5	8.3880	.0342	A	10	8.3820	.0290	.35
LAB- 1	(AA)	5	8.3440	.0134	5	8.3200	.0000	***R**	10	8.3320	.0155	.19
LAB- 2	(AA)	5	8.2340	.0305	5	8.2480	.0327	A	10	8.2410	.0307	.37
LAB- 5	(TITR)	5	8.5960	.0251	5	8.5960	.0134	A	10	8.5960	.0190	.22
LAB- 6	(TITR)	5	8.4020	.0415	5	8.3800	.0158	A	10	8.3910	.0318	.38
LAB- 9	(TITR)	5	8.6200	.0500	5	8.5500	.0274	REJECT	10	8.5850	.0530	.62
LAB-13	(TITR)	5	8.3717	.0141	5	8.3657	.0089	A	10	8.3687	.0116	.14
LAB-14	(TITR)	5	8.5460	.0134	5	8.4900	0.0000	***R**	10	8.5180	.0308	.36
LAB-18	(TITR)	6	8.4983	.0412	6	8.5350	.0464	A	12	8.5167	.0460	.54
LAB-19	(AA)	5	8.9280	.0390	5	8.9360	.0358	A	10	8.9320	.0355	.40
LAB-21	(TITR)	5	8.4040	.0270	5	8.3980	.0110	A	10	8.4010	.0197	.23
LAB-23	(TITR)	5	8.4300	.0224	5	8.3900	.0224	REJECT	10	8.4100	.0298	.35
LAB-24	(TITR)	5	8.3520	.0045	5	8.3500	.0000	A	10	8.3510	.0032	.04
LAB-26	(TITR)	5	8.5940	.0134	5	8.5820	.0164	A	10	8.5880	.0155	.18
LAB-29	(TITR)	5	8.4240	.0602	5	8.4680	.0920	A	10	8.4460	.0769	.91
LAB-30	(TITR)	5	8.1600	.0548	5	8.1200	.0447	A	10	8.1400	.0516	.63
LAB-31	(TITR)	5	8.6800	.0300	5	8.6620	.0268	A	10	8.6710	.0285	.33
LAB-32	(AA)	5	9.1360	.0546	5	9.1000	.0791	A	10	9.1180	.0668	.73
LAB-34	(TITR)	5	8.4480	.0110	5	8.4480	.0110	A	10	8.4480	.0103	.12
LAB-35	(TITR)	2	8.3250	.0354	2	8.3250	.0071	A	4	8.3250	.0208	.25
LAB-38	(AA)	5	8.3980	.0567	5	8.2480	.1987	A	10	8.3230	.1588	1.91
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	8.1660	.2007	2.46
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	7.9660	.0261	.33
LAB-40	(AA)	6	8.2167	.0408	6	8.2083	.0585	A	12	8.2125	.0483	.59

Variance between sets, between bottles and within bottles = 1.54×10^{-2}, 5.99×10^{-4} and 2.23×10^{-3}, respectively.

Hg (µg/g)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 2	(AA)	5	6.1600	.3782	5	6.1400	.3050	A	10	6.1500	.3240	5.27
LAB- 5	(AA)	5	5.7800	.0837	5	5.7000	.1225	A	10	5.7400	.1075	1.87
LAB- 6	(AA)	5	5.1400	.2510	5	5.1600	.1517	A	10	5.1500	.1958	3.80
LAB- 9	(AA)	5	5.8000	.4472	5	6.0000	0.0000	A	10	5.9000	.3162	5.36
LAB-14	(AA)	5	4.8200	.1789	5	4.6000	.1000	REJECT	10	4.7100	.1792	3.80
LAB-18	(AA)	4	6.6000	.5477	4	6.2500	.4041	A	8	6.4250	.4833	7.52
LAB-23	(AA)	5	7.0000	.1414	5	6.5600	.1342	REJECT	10	6.7800	.2658	3.92
LAB-24	(AA)	5	6.4000	1.1402	5	6.6000	.5477	A	10	6.5000	.8498	13.07
LAB-30	(AA)	5	5.2000	.0000	5	5.2800	.1095	A	10	5.2400	.0843	1.61
LAB-34	(COLOR)	5	4.7860	.1085	5	4.9660	.1172	REJECT	10	4.8760	.1426	2.92
LAB-35	(AA)	2	5.5000	.7071	2	4.0000	0.0000	A	4	4.7500	.9574	20.16
LAB-38	(AA)	5	4.4820	.1105	5	4.3720	.2357	A	10	4.4270	.1830	4.13
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	4.8000	2.1679	45.17
LAB-40	(AA)	5	4.5260	.5382	5	4.4100	.3489	A	10	4.4680	.4319	9.67

Variance between sets, between bottles and within bottles = 6.58×10^{-1}, 0 and 1.25×10^{-1}, respectively.

TABLE 7 (cont'd)

Pb (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(TITR)	5	65.1680	.0228	5	64.9000	.0992	REJECT	10	65.0340	.1567	.24
LAB- 1	(TITR)	5	64.5640	.1454	5	64.5200	.0875	A	10	64.5420	.1155	.18
LAB- 2	(AA)	5	64.8600	.1430	5	64.9840	.1632	A	10	64.9220	.1587	.24
LAB- 5	(TITR)	5	65.1580	.0349	5	64.8620	.0540	REJECT	10	65.0100	.1618	.25
LAB- 6	(TITR)	5	64.4660	.2094	5	64.4100	.2120	A	10	64.4380	.2008	.31
LAB- 9	(TITR)	5	65.0800	.1095	5	65.0000	.2449	A	10	65.0400	.1838	.28
LAB-13	(TITR)	5	65.0110	.0548	5	65.0110	.0548	A	10	65.0110	.0517	.08
LAB-14	(TITR)	5	65.1900	.0837	5	65.1500	.0447	A	10	65.1700	.0667	.10
LAB-16	(TITR)	5	64.6620	.0826	5	64.5980	.0377	A	10	64.6300	.0693	.11
LAB-18	(TITR)	6	64.9367	.0686	6	64.8950	.0764	A	12	64.9158	.0725	.11
LAB-19	(AA)	5	64.7400	.2408	5	64.6900	.2247	A	10	64.7150	.2212	.34
LAB-21	(TITR)	5	64.9040	.0416	5	64.9120	.0432	A	10	64.9080	.0402	.06
LAB-23	(TITR)	5	64.6600	.1673	5	64.6400	.0548	A	10	64.6500	.1179	.18
LAB-24	(GRAV)	5	65.4200	.0831	5	65.4100	.0510	A	10	65.4150	.0652	.10
LAB-26	(AA)	5	64.6000	.1414	5	64.6000	.1414	A	10	64.6000	.1333	.21
LAB-26	(TITR)	5	64.1860	.2107	5	64.0000	.0809	A	10	64.0930	.1796	.28
LAB-29	(GRAV)	5	64.2480	.0110	5	64.2380	.0396	A	10	64.2430	.0279	.04
LAB-30	(TITR)	5	64.9400	.1095	5	64.8800	.0447	A	10	64.9100	.0850	.13
LAB-31	(GRAV)	5	63.6500	.2182	4	63.6725	.1597	A	9	63.6600	.1830	.29
LAB-32	(TITR)	5	64.6120	.1287	5	64.5700	.0866	A	10	64.5910	.1058	.16
LAB-33	(GRAV)	5	64.5260	.0537	5	64.5380	.0438	A	10	64.5320	.0466	.07
LAB-34	(TITR)	5	64.9780	.1494	5	64.7720	.0807	REJECT	10	64.8750	.1569	.24
LAB-35	(TITR)	6	64.7317	.0783	6	64.8450	.0758	REJECT	12	64.7883	.0944	.15
LAB-37	(TITR)	5	64.6120	.0893	5	64.6200	.0520	A	10	64.6160	.0690	.11
LAB-38	(GRAV)	5	64.4420	.2202	5	64.2580	.2295	A	10	64.3500	.2331	.36
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	64.2000	.1393	.22
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	61.8580	.2975	.48
LAB-40	(TITR)	10	64.8100	.2079	9	64.7444	.1509	A	19	64.7789	.1813	.28
LAB-45	(TITR)	5	64.6660	.0261	5	64.6260	.0336	A	10	64.6460	.0353	.05

Variance between sets, between bottles and within bottles = 8.45×10^{-2}, 3.58×10^{-3} and 1.45×10^{-2}, respectively.

S (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 5	(GRAV)	5	18.0620	.0522	5	17.9840	.0251	REJECT	10	18.0230	.0564	.31
LAB- 6	(GRAV)	5	17.5340	.0422	5	17.6540	.0654	REJECT	10	17.5940	.0818	.47
LAB- 9	(GRAV)	5	17.5620	.0409	5	17.5720	.0295	A	10	17.5670	.0340	.19
LAB-13	(GRAV)	5	17.9572	.0433	5	17.9211	.0655	A	10	17.9391	.0557	.31
LAB-14	(COMB)	5	17.6500	.2208	5	17.6500	.0612	A	10	17.6500	.1528	.87
LAB-14	(GRAV)	5	17.9220	.0638	5	17.8860	.0999	A	10	17.9040	.0813	.45
LAB-18	(COMB)	4	17.8375	.0640	4	17.8875	.0250	A	8	17.8625	.0523	.29
LAB-21	(GRAV)	5	17.9740	.1216	5	18.0380	.1062	A	10	18.0060	.1128	.63
LAB-23	(GRAV)	5	17.8120	.0942	5	17.6940	.0329	REJECT	10	17.7530	.0910	.51
LAB-26	(COMB)	5	18.2400	.1673	5	18.2600	.1673	A	10	18.2500	.1581	.87
LAB-30	(GRAV)	5	16.9200	.3271	5	16.7600	.1817	A	10	16.8400	.2633	1.56
LAB-31	(GRAV)	5	17.6080	.1326	5	17.7180	.1035	A	10	17.6630	.1262	.71
LAB-34	(GRAV)	5	17.6520	.0363	5	17.6800	.0938	A	10	17.6660	.0687	.39
LAB-35	(GRAV)	2	17.9150	.2051	2	17.8150	.0354	A	4	17.8650	.1333	.75
LAB-38	(GRAV)	5	18.0340	.0472	4	18.0425	.0340	A	9	18.0378	.0396	.22
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	18.0520	.0522	.29
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	18.8560	.1967	1.04

Variance between sets, between bottles and within bottles = 4.66×10^{-2}, 5.21×10^{-4} and 9.78×10^{-3}, respectively.

Sb (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 3	(COLOR)	5	.3708	.0049	5	.3684	.0050	A	10	.3696	.0048	1.31
LAB- 5	(AA)	5	.3660	.0055	5	.3660	.0055	A	10	.3660	.0052	1.41
LAB- 6	(AA)	5	.4004	.0056	5	.3996	.0042	A	10	.4000	.0047	1.17
LAB- 9	(AA)	5	.3280	.0110	5	.3384	.0148	A	10	.3332	.0135	4.04
LAB-14	(AA)	5	.3682	.0059	5	.3660	.0030	A	10	.3671	.0046	1.24
LAB-19	(AA)	5	.2974	.0021	5	.2988	.0008	A	10	.2981	.0017	.56
LAB-21	(AA)	5	.3424	.0005	5	.3478	.0008	REJECT	10	.3451	.0029	.85
LAB-23	(COLOR)	5	.3488	.0166	5	.3544	.0171	A	10	.3516	.0162	4.60
LAB-24	(AA)	5	.3696	.0054	5	.3750	.0024	A	10	.3723	.0049	1.30
LAB-30	(AA)	5	.3560	.0055	5	.3600	0.0000	A	10	.3580	.0042	1.18
LAB-34	(AA)	5	.3310	.0061	5	.3316	.0030	A	10	.3313	.0045	1.37
LAB-35	(AA)	2	.3850	.0071	2	.3850	.0071	A	4	.3850	.0058	1.50
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.3254	.0102	3.15
LAB-40	(ES)	5	.3300	.0224	5	.3240	.0182	A	10	.3270	.0195	5.95

Variance between sets, between bottles and within bottles = 4.66×10^{-4}, 0 and 9.33×10^{-5}, respectively.

TABLE 7 (cont'd)

SiO_2 (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(GRAV)	5	.7300	.0141	5	.6800	.0000	***R**	10	.7050	.0280	3.97
LAB- 2	(AA)	5	.7268	.0047	5	.7260	.0106	A	10	.7264	.0077	1.06
LAB- 4	(GRAV)	5	.7188	.0067	5	.7120	.0038	A	10	.7154	.0063	.88
LAB- 5	(GRAV)	5	.6480	.0164	5	.6340	.0114	A	10	.6410	.0152	2.38
LAB- 9	(GRAV)	5	1.0900	.0548	5	1.1100	.0548	A	10	1.1000	.0527	4.79
LAB-13	(GRAV)	5	.7570	.0336	5	.8070	.0152	REJECT	10	.7820	.0360	4.61
LAB-14	(GRAV)	5	.8040	.0192	5	.8682	.0743	A	10	.8361	.0614	7.34
LAB-18	(GRAV)	4	.5450	.0191	4	.5925	.0299	REJECT	8	.5688	.0344	6.05
LAB-19	(GRAV)	5	.7446	.0364	5	.7614	.0331	A	10	.7530	.0340	4.51
LAB-21	(GRAV)	5	.5980	.0045	5	.5860	.0114	A	10	.5920	.0103	1.74
LAB-23	(GRAV)	5	.7980	.0192	5	.8060	.0134	A	10	.8020	.0162	2.02
LAB-24	(GRAV)	5	.7480	.0482	5	.7500	.0235	A	10	.7490	.0357	4.77
LAB-26	(AA)	5	.7460	.0513	5	.7680	.0554	A	10	.7570	.0517	6.82
LAB-29	(GRAV)	5	.7120	.0179	5	.6800	.0490	A	10	.6960	.0386	5.55
LAB-34	(GRAV)	5	.7600	.0122	5	.7540	.0114	A	10	.7570	.0116	1.53
LAB-35	(GRAV)	4	.6425	.0222	4	.6525	.0287	A	8	.6475	.0243	3.76
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	.4380	.1588	36.26
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	1.0560	.0365	3.45

Variance between sets, between bottles and within bottles = 2.43×10^{-3}, 2.81×10^{-4} and 9.41×10^{-4}, respectively.

Zn (wt %)

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(AA)	5	4.4500	.0187	5	4.4420	.0303	A	10	4.4460	.0241	.54
LAB- 2	(AA)	5	4.4280	.0286	5	4.4460	.0114	A	10	4.4370	.0226	.51
LAB- 4	(TITR)	5	4.4660	.0230	5	4.4460	.0207	A	10	4.4560	.0232	.52
LAB- 5	(TITR)	5	4.3160	.0261	4	4.2400	0.0000	***R**	9	4.2822	.0441	1.03
LAB- 6	(AA)	5	4.4120	.0217	5	4.4000	.0173	A	10	4.4060	.0196	.44
LAB- 9	(AA)	5	4.2920	.0363	5	4.2740	.0152	A	10	4.2830	.0279	.65
LAB-13	(TITR)	5	4.4482	.0205	5	4.4362	.0174	A	10	4.4422	.0190	.43
LAB-14	(TITR)	5	4.5600	.0000	5	4.5480	.0164	A	10	4.5540	.0126	.28
LAB-16	(AA)	5	4.3060	.0251	5	4.2900	.0332	A	10	4.2980	.0290	.67
LAB-18	(TITR)	5	4.6580	.0409	6	4.6600	.0610	A	11	4.6591	.0503	1.08
LAB-19	(AA)	5	4.4940	.0251	5	4.4960	.0167	A	10	4.4950	.0201	.45
LAB-21	(AA)	5	4.3200	.0122	5	4.3940	.0219	REJECT	10	4.3570	.0424	.97
LAB-23	(AA)	5	4.5400	.0548	5	4.5200	.0447	A	10	4.5300	.0483	1.07
LAB-24	(TITR)	5	4.5600	.0620	5	4.5920	.0444	A	10	4.5760	.0536	1.17
LAB-24	(AA)	5	4.4660	.0241	5	4.5140	.0279	REJECT	10	4.4900	.0353	.79
LAB-26	(AA)	5	4.4400	.0583	5	4.4240	.0792	A	10	4.4320	.0661	1.49
LAB-30	(TITR)	5	4.4220	.0110	5	4.4060	.0152	A	10	4.4140	.0151	.34
LAB-31	(AA)	5	4.3340	.0219	5	4.3500	.0381	A	10	4.3420	.0305	.70
LAB-32	(AA)	5	5.0600	.0224	5	5.0500	.0612	A	10	5.0550	.0438	.87
LAB-33	(TITR)	3	4.3267	.0115	3	4.3933	.0987	A	6	4.3600	.0727	1.67
LAB-33	(TITR)	2	4.5050	.0636	2	4.4700	0.0000	A	4	4.4875	.0419	.93
LAB-34	(AA)	4	4.4550	.0755	5	4.3980	.0444	A	9	4.4233	.0634	1.43
LAB-35	(AA)	4	4.3850	.0858	4	4.3600	.0942	A	8	4.3725	.0845	1.93
LAB-38	(AA)	5	4.3320	.0396	5	4.2880	.0602	A	10	4.3100	.0533	1.24
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	4.4240	.1467	3.32
LAB-39	(AA)	THERE IS ONLY 1 BOTTLE							5	4.2560	.0921	2.16
LAB-40	(AA)	6	4.3267	.1127	6	4.3050	.1084	A	12	4.3158	.1060	2.46
LAB-45	(TITR)	5	4.4460	.0055	5	4.4500	.0071	A	10	4.4480	.0063	.14
LAB-45	(TITR)	5	4.4100	.0071	5	4.4260	.0089	REJECT	10	4.4180	.0114	.26

Variance between sets, between bottles and within bottles = 9.22×10^{-3}, 0 and 1.77×10^{-3}, respectively.

TABLE 8

Analytical results for reference concentrate CPB-1†

Ag (μg/g)

LAB-19 (AA)	646	635	637	641	650	646	644	642	642	645
LAB-24 (AA)	615	613	615	615	613	618	615	608	612	615
LAB-26 (AA)	628	626	634	632	624	632	626	632	630	622
LAB-30 (AA)	607	603	613	607	626	618	619	617	624	626
LAB-31 (AA)	630	620	620	610	610	620	630	620	620	620
LAB-33 (AA)	624	630	629	624	626	630	627	626	627	623
LAB-35 (AA)	634	636	638	637						
LAB-38 (AA)	630	620	610	610	630	600	610	610	610	600
LAB-40 (AA)	625	655	650	670	640	650	655	650	660	645
LAB-39 (ES)	640	610	645	630	650					
LAB- 5 (FA)	614	610	607	609	612	612	607	613	610	606
LAB- 6 (FA)	630	627	630	623	627	626	614	617	628	621
LAB-14 (FA)	628	618	624	618	632	623	618	618	618	624
LAB-18 (FA)	640	660	640	640	640	660	660	640		
LAB-21 (FA)	640	641	632	638	636	622	628	627	627	628
LAB-34 (FA)	625	637	622	628	629	620	633	630	636	630
LAB-37 (FA)	633	629	628	633	628	629	629	628	628	633
LAB- 1 (FA-AA)	604	604	617	624	610	631	624	624	631	631
LAB- 9 (FA-AA)	605	598	604	595	597	596				
LAB-12 (FA-AA)	645	644	644	641	638	640	643	640	641	638
LAB-12 (FA-AA)	628	634	638	629	635	628	629	630	638	645
LAB-16 (FA-AA)	600	606	605	602	602	612	600	595	598	595

Al_2O_3 (wt %)

LAB- 1 (AA)	.34	.34	.34	.32	.36	.30	.34	.30	.30	.34
LAB- 4 (AA)	.29	.28	.28	.29	.30	.29	.28	.29	.29	.29
LAB- 5 (AA)	.28	.28	.28	.28	.28	.27	.28	.28	.28	.28
LAB- 6 (AA)	.28	.29	.28	.29	.28	.30	.29	.29	.29	.30
LAB- 9 (AA)	.30	.30	.28	.30	.30	.26	.28	.28	.26	.28
LAB-14 (AA)	.28	.28	.28	.28	.28	.28	.28	.28	.28	.28
LAB-18 (AA)	.30	.32	.31	.29	.32	.30	.29	.30		
*LAB-19 (AA)	.18	.19	.19	.19	.19	.20	.20	.19	.19	.20
*LAB-21 (AA)	.34	.35	.36	.36	.35	.36	.34	.35	.34	.32
LAB-26 (AA)	.28	.26	.25	.26	.28	.25	.27	.27	.27	.29
LAB-23 (AA)	.27	.27	.26	.26	.26	.25	.25	.25	.26	.25
	.27	.26	.26	.26	.27	.25	.25	.25	.27	.27
LAB-34 (AA)	.29	.28	.28	.29	.28	.29	.29	.29	.29	.29
*LAB-35 (AA)	.17	.18	.16	.18						

As (wt %)

LAB-16 (AA)	.049	.051	.051	.049	.049	.050	.052	.049	.049	.050
LAB-30 (AA)	.063	.066	.066	.066	.064	.066	.065	.066	.066	.066
LAB-38 (AA)	.057	.061	.058	.060	.057	.063	.060	.060	.063	.058
*LAB-13 (TITR)	.182	.178	.182	.182	.182	.182	.182	.184	.178	.184
LAB-21 (TITR)	.045	.049	.042	.052	.053	.049	.050	.050	.054	.051
LAB-33 (TITR)	.051	.058	.061	.057	.051	.061	.061	.055	.061	.060
LAB-35 (TITR)	.050	.050	.040	.040						
LAB- 3 (COLOR)	.061	.062	.061	.062	.061	.062	.062	.062	.061	.062
LAB- 5 (COLOR)	.050	.050	.040	.040	.040	.040	.040	.040	.040	.040
*LAB- 6 (COLOR)	.090	.087	.087	.092	.087	.088	.087	.096	.084	.087
LAB- 9 (COLOR)	.057	.056	.068	.056	.065	.068	.066	.065	.061	.059
LAB-18 (COLOR)	.062	.060	.057	.055	.060	.060	.056	.057		
LAB-23 (COLOR)	.057	.062	.070	.060	.064	.068	.064	.062	.053	.064
	.060	.071	.064	.050	.036	.058	.058	.057	.038	.046
	.053	.055								
LAB-24 (COLOR)	.056	.057	.067	.057	.055	.051	.059	.057	.070	.057
LAB-34 (COLOR)	.047	.049	.049	.043	.044	.041	.042	.045	.041	.045
LAB-40 (COLOR)	.059	.059	.056	.059	.059	.061	.060	.057	.059	.057
LAB-39 (ES)	.066	.060	.063	.069	.067					
LAB-24 (NAA)	.050	.047	.050	.046	.050	.052	.052	.049	.050	.045
	.046	.046								
LAB-14	.059	.060	.059	.059	.059	.060				

†See legend and note at end of table on p 28.

TABLE 8 (cont'd)

Au (μg/g)

LAB- 5 (FA)	.10	.10	.10	.10	.10	.10	.10	.10	.10	.10
LAB- 1 (FA-AA)	.27	.21	.27	.27	.21	.21	.21	.24	.21	.21
	.17	.21	.21	.21	.17	.21	.24	.21	.24	.24
	.24	.24	.21	.21	.24					
LAB-14 (FA-AA)	.05	.05	.05	.06	.06	.06	.06	.06	.05	.06
LAB-18 (FA-AA)	.31	.27	.27	.25	.25	.27	.27	.30		

Bi (wt %)

*LAB- 1 (AA)	.029	.029	.029	.026	.029	.032	.029	.029	.030	.028
LAB- 5 (AA)	.020	.020	.020	.020	.020	.020	.020	.020	.020	.020
LAB- 6 (AA)	.021	.021	.020	.021	.022	.022	.021	.021	.021	.022
*LAB- 9 (AA)	.035	.034	.034	.033	.034	.034	.030	.031	.032	.030
*LAB-13 (AA)	.331	.331	.331	.300	.331	.331	.331	.331	.331	.300
LAB-14 (AA)	.019	.021	.020	.018	.017	.021				
LAB-19 (AA)	.022	.022	.022	.022	.022	.022	.022	.022	.022	.022
LAB-21 (AA)	.025	.025	.025	.025	.025	.025	.025	.025	.025	.025
LAB-23 (AA)	.023	.023	.024	.024	.024	.024	.023	.024	.023	.023
	.024	.024	.025	.024	.023	.024	.024	.023	.024	.024
LAB-30 (AA)	.024	.024	.024	.024	.023	.024	.023	.023	.023	.024
LAB-34 (AA)	.021	.021	.022	.021	.021	.021	.021	.021	.021	.021
LAB-35 (AA)	.023	.024	.022	.024	.025	.022	.020	.024	.022	.021
	.025	.022								
LAB-38 (AA)	.025	.025	.024	.024	.024	.024	.024	.024	.025	.025
LAB- 3 (COLOR)	.022	.021	.022	.022	.022	.022	.021	.021	.022	.021
LAB-24 (COLOR)	.028	.028	.026	.026	.026	.024	.025	.027	.029	.027
LAB-18 (ES)	.020	.021	.020	.018	.020	.018	.018	.018		
LAB-39 (ES)	.025	.026	.023	.020	.022					
LAB-40 (ES)	.022	.025	.023	.020	.022	.022	.024	.024	.020	.022

CaO (wt %)

LAB- 1 (AA)	.95	.94	.95	.94	.94	.95	.99	.98	.94	.97
LAB- 4 (AA)	.88	.88	.87	.87	.88	.89	.87	.88	.88	.88
LAB- 5 (AA)	.82	.82	.85	.83	.85	.83	.83	.83	.84	.84
LAB- 6 (AA)	.89	.88	.89	.88	.86	.89	.89	.88	.88	.86
LAB- 9 (AA)	.90	.90	.88	.89	.91	.91	.89	.89	.89	.88
*LAB-13 (AA)	.53	.53	.53	.53	.53	.53	.53	.53	.53	.53
LAB-14 (AA)	.82	.82	.82	.82	1.13*	.81	.81	.87	.90	.90
*LAB-18 (AA)	1.08	1.08	1.08	1.09	.98	1.03	1.03	1.03		
*LAB-19 (AA)	.84	.83	.83	.83	.83	.83	.83	.83	.83	.83
*LAB-21 (AA)	1.04	1.04	1.04	1.05	1.05	1.06	1.07	1.08	1.07	1.05
LAB-26 (AA)	.82	.81	.81	.81	.77	.81	.78	.80	.82	.79
LAB-23 (AA)	.85	.85	.84	.84	.83	.89	.86	.89	.87	.88
	.83	.84	.84	.84	.84	.93	.94	.91	.89	.85
*LAB-30 (AA)	.80	.81	.80	.80	.81	.80	.80	.81	.80	.81
LAB-34 (AA)	.97	.92	.94	.95	.96	.94	.94	.95	.95	.93
*LAB-35 (AA)	.86	.87	.88	.88	.88	.90	.88	.90		
*LAB-38 (AA)	.83	.84	.84	.83	.84	.82	.81	.83	.82	.83

Cd (wt %)

LAB- 1 (AA)	.015	.015	.015	.015	.015	.015	.015	.015	.015	.015
LAB- 5 (AA)	.015	.015	.015	.015	.016	.016	.015	.015	.015	.015
LAB- 6 (AA)	.014	.014	.014	.014	.014	.014	.014	.014	.014	.014
LAB- 9 (AA)	.013	.013	.014	.013	.014	.015	.015	.014	.015	.015
*LAB-13 (AA)	.010	.010	.010	.010	.010	.010	.010	.010	.010	.010
LAB-14 (AA)	.014	.015	.014	.015	.015	.015	.014	.014	.015	.015
LAB-18 (AA)	.014	.014	.014	.014	.014	.014				
LAB-19 (AA)	.015	.015	.015	.015	.015	.015	.015	.015	.015	.015
LAB-21 (AA)	.014	.014	.014	.015	.015	.014	.014	.014	.014	.014
LAB-23 (AA)	.012	.012	.013	.012	.012	.014	.014	.014	.014	.014
	.012	.012	.012	.012	.012	.014	.014	.014	.014	.014
*LAB-30 (AA)	.020	.020	.019	.016	.019	.016	.019	.019	.019	.019
LAB-34 (AA)	.015	.014	.015	.015	.014	.015	.015	.015	.015	.015
LAB-35 (AA)	.013	.014	.013	.014	.013	.014	.013	.014	.014	.014
	.014	.014								
LAB-38 (AA)	.015	.015	.015	.015	.014	.015	.015	.015	.015	.015
LAB-40 (AA)	.016	.015	.016	.016	.017	.015	.016	.015	.016	.016
	.016	.015								
*LAB-39 (ES)	.016	.013	.017	.019	.015					

TABLE 8 (cont'd)

Cu (wt %)

LAB- 1 (AA)	.260	.260	.260	.260	.260	.260	.260	.260	.260	.260
LAB- 1 (AA)	.260	.260	.260	.270	.260	.260	.260	.270	.260	.260
LAB- 2 (AA)	.249	.248	.251	.250	.254	.253	.256	.251	.256	.254
LAB- 5 (AA)	.240	.240	.240	.240	.240	.240	.240	.240	.240	.240
LAB- 6 (AA)	.245	.245	.244	.245	.244	.245	.245	.245	.240	.240
LAB- 9 (AA)	.241	.230	.227	.241	.228	.248	.244	.242	.257	.255
LAB-14 (AA)	.244	.244	.248	.243	.242	.249	.244	.245	.244	.244
LAB-18 (AA)	.260	.270	.260	.250	.260	.260	.270	.260		
LAB-19 (AA)	.258	.256	.257	.259	.259	.256	.260	.260	.261	.260
LAB-21 (AA)	.248	.249	.248	.251	.248	.248	.246	.247	.247	.246
LAB-23 (AA)	.260	.260	.260	.260	.260	.260	.260	.260	.260	.260
	.260	.260	.260	.260	.260	.260	.260	.280	.290*	.260
LAB-24 (AA)	.250	.250	.250	.240	.250	.240	.250	.250	.250	.240
LAB-26 (AA)	.252	.255	.253	.255	.255	.251	.256	.253	.256	.254
LAB-30 (AA)	.249	.253	.253	.252	.256	.253	.251	.245	.245	.246
LAB-31 (AA)	.260	.270	.260	.260	.260	.270	.260	.260	.260	.260
LAB-32 (AA)	.273	.273	.265	.270	.273	.265	.270	.268	.268	.268
LAB-34 (AA)	.250	.248	.244	.251	.248	.247	.247	.248	.246	.247
LAB-35 (AA)	.260	.280	.250	.250	.260	.260	.260	.270	.250	.270
	.260	.240								
LAB-38 (AA)	.248	.252	.246	.241	.250	.243	.238	.247	.243	.240
LAB-40 (AA)	.240	.248	.247	.248	.249	.250	.240	.248	.247	.249
	.250	.250								
* LAB-39 (TITR)	.240	.230	.200	.230	.200					
* LAB-39 (TITR)	.130	.130	.130	.140	.130					
LAB- 1 (COLOR)	.251	.261	.259	.259	.252	.255	.255	.256	.260	.263
LAB- 3 (COLOR)	.263	.266	.265	.268	.272	.266	.268	.264	.266	.266

Fe (wt %)

LAB- 1 (AA)	8.35	8.35	8.32	8.35	8.35	8.32	8.32	8.32	8.32	8.32
LAB- 2 (AA)	8.20	8.27	8.21	8.26	8.23	8.25	8.21	8.28	8.28	8.22
* LAB-19 (AA)	8.96	8.86	8.94	8.94	8.94	8.88	8.96	8.96	8.96	8.92
* LAB-32 (AA)	9.05	9.15	9.15	9.20	9.13	9.20	9.15	9.05	9.00	9.10
LAB-38 (AA)	8.42	8.43	8.36	8.32	8.46	8.42	8.05	8.49	8.20	8.08
LAB-40 (AA)	8.20	8.20	8.25	8.25	8.15	8.25	8.20	8.25	8.10	8.25
	8.25	8.20								
LAB- 1 (TITR)	8.34	8.40	8.37	8.40	8.37	8.43	8.34	8.40	8.37	8.40
LAB- 5 (TITR)	8.59	8.56	8.62	8.59	8.62	8.59	8.62	8.59	8.59	8.59
LAB- 6 (TITR)	8.43	8.44	8.42	8.34	8.38	8.39	8.40	8.37	8.38	8.36
LAB- 9 (TITR)	8.67	8.57	8.67	8.57	8.62	8.57	8.52	8.57	8.52	8.57
LAB-13 (TITR)	8.38	8.36	8.36	8.36	8.39	8.38	8.36	8.36	8.36	8.36
LAB-14 (TITR)	8.57	8.54	8.54	8.54	8.54	8.49	8.49	8.49	8.49	8.49
LAB-18 (TITR)	8.43	8.53	8.53	8.53	8.47	8.50	8.53	8.60	8.48	8.58
	8.50	8.52								
LAB-21 (TITR)	8.45	8.40	8.40	8.38	8.39	8.40	8.38	8.40	8.40	8.41
LAB-23 (TITR)	8.42	8.42	8.47	8.42	8.42	8.35	8.40	8.40	8.40	8.40
LAB-24 (TITR)	8.36	8.35	8.35	8.35	8.35	8.35	8.35	8.35	8.35	8.35
LAB-26 (TITR)	8.60	8.60	8.60	8.57	8.60	8.57	8.57	8.57	8.60	8.60
LAB-29 (TITR)	8.38	8.38	8.38	8.49	8.49	8.38	8.38	8.49	8.49	8.60
* LAB-30 (TITR)	8.10	8.20	8.20	8.20	8.10	8.10	8.10	8.10	8.10	8.20
LAB-31 (TITR)	8.69	8.63	8.68	8.71	8.69	8.66	8.62	8.66	8.69	8.68
LAB-34 (TITR)	8.44	8.44	8.44	8.46	8.46	8.46	8.44	8.44	8.44	8.46
LAB-35 (TITR)	8.30	8.35	8.32	8.33						
* LAB-39 (TITR)	8.10	8.10	8.40	8.33	7.90					
* LAB-39 (TITR)	7.98	7.92	7.97	7.98	7.98					

Hg (µg/g)

LAB- 2 (AA)	5.9	6.8	6.2	6.0	5.9	6.2	6.5	6.3	6.0	5.7
LAB- 5 (AA)	5.7	5.8	5.8	5.9	5.7	5.9	5.7	5.6	5.7	5.6
LAB- 6 (AA)	5.4	4.9	4.9	5.4	5.1	5.1	5.2	5.0	5.4	5.1
LAB- 9 (AA)	6.0	5.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
LAB-14 (AA)	5.0	5.0	4.8	4.7	4.6	4.7	4.7	4.6	4.5	4.5
LAB-18 (AA)	6.7	6.9	5.8	7.0	6.0	6.8	5.9	6.3		
LAB-23 (AA)	6.8	6.9	7.1	7.1	7.1	6.5	6.7	6.7	6.5	6.4
LAB-24 (AA)	6.0	6.0	5.0	7.0	8.0	6.0	7.0	7.0	6.0	7.0
LAB-30 (AA)	5.2	5.2	5.2	5.2	5.2	5.4	5.2	5.2	5.2	5.4
LAB-35 (AA)	5.0	6.0	4.0	4.0						
LAB-38 (AA)	4.5	4.5	4.3	4.6	4.6	4.7	4.4	4.0	4.3	4.5
LAB-40 (AA)	5.4	4.4	4.0	4.6	4.3	4.8	4.3	4.0	4.8	4.3
LAB-34 (COLOR)	4.9	4.8	4.8	4.6	4.8	5.0	5.0	5.0	4.8	5.1
LAB-39 (ES)	7.0	7.0	2.0	4.0	4.0					

TABLE 8 (cont'd)

In (μg/g)

LAB- 6 (AA)	17	17	20	17	20	17	17	17	17	20
*LAB-18 (AA)	22	22	22	24						
LAB-34 (AA)	13	16	12	14	13	12	14	12	12	11
LAB-35 (AA)	12	12	13	14	14	11	10	11		
LAB-23 (COLOR)	14	10	12	12	15	11	12	11	12	14
LAB-39 (ES)	15	20	20	15	15					
LAB-40 (ES)	14	11	11	12	12	13	11	11	10	12

MgO (wt %)

LAB- 1 (AA)	.16	.16	.16	.16	.17	.18	.17	.17	.17	.17
LAB- 4 (AA)	.17	.16	.15	.16	.16	.16	.16	.16	.16	.16
LAB- 5 (AA)	.16	.16	.16	.16	.16	.16	.16	.16	.16	.16
LAB- 6 (AA)	.14	.14	.15	.14	.14	.14	.14	.14	.15	.14
LAB- 9 (AA)	.17	.17	.17	.18	.17	.17	.17	.17	.17	.17
*LAB-13 (AA)	.10	.11	.11	.11	.10	.10	.11	.11	.11	.11
LAB-14 (AA)	.15	.15	.16	.16	.15	.15	.16	.16	.16	.16
LAB-18 (AA)	.16	.16	.17	.16	.16	.16	.16	.16		
*LAB-19 (AA)	.15	.15	.15	.15	.15	.15	.15	.15	.15	.15
LAB-21 (AA)	.17	.17	.17	.17	.18	.17	.17	.17	.17	.17
LAB-23 (AA)	.14	.15	.13	.16	.13	.13	.13	.13	.14	.14
	.16	.14	.13	.13	.13	.13	.14	.13	.13	.13
LAB-26 (AA)	.14	.14	.14	.14	.14	.14	.14	.14	.14	.14
*LAB-30 (AA)	.11	.10	.10	.10	.11	.10	.10	.10	.10	.11
LAB-34 (AA)	.15	.15	.15	.15	.15	.15	.15	.15	.15	.15
*LAB-35 (AA)	.15	.14	.15	.14	.15	.14	.16	.14		
*LAB-38 (AA)	.11	.11	.10	.10	.10	.09	.10	.11	.10	.14*

Mn (wt %)

LAB- 5 (AA)	.036	.036	.036	.037	.037	.035	.037	.037	.037	.037
LAB- 6 (AA)	.040	.042	.042	.042	.040	.040	.040	.040	.042	.040
LAB- 9 (AA)	.039	.040	.041	.039	.040	.041	.041	.041	.040	.041
LAB-14 (AA)	.038	.038	.037	.037	.037	.037	.037	.037	.037	.038
LAB-18 (AA)	.042	.042	.042	.042	.042	.042	.042	.042		
LAB-19 (AA)	.038	.037	.037	.037	.037	.037	.037	.038	.038	.037
LAB-21 (AA)	.043	.044	.043	.043	.043	.044	.043	.043	.043	.042
LAB-23 (AA)	.042	.042	.039	.042	.040	.040	.040	.040	.040	.040
	.042	.040	.040	.040	.042	.040	.040	.040	.042	.040
LAB-34 (AA)	.040	.040	.041	.040	.039	.040	.040	.039	.040	.039
LAB-35 (AA)	.029	.029	.037	.037	.035	.036	.029	.030	.034	.037
	.035	.036								
LAB-38 (AA)	.032	.032	.032	.033	.032	.032	.032	.032	.032	.032
LAB-40 (AA)	.045	.040	.040	.045	.040	.040	.045	.043	.040	.044
	.041	.040								
LAB- 3 (COLOR)	.039	.040	.040	.039	.040	.039	.040	.039	.039	.040
*LAB-39 (ES)	.047	.050	.044	.048	.055					

TABLE 8 (cont'd)

Pb (wt %)

LAB- 2 (AA)	64.85	65.07	64.72	64.74	64.92	64.74	65.18	64.98	64.95	65.07
LAB-19 (AA)	64.90	64.60	64.40	64.80	65.00	64.40	64.85	64.80	64.50	64.90
LAB-26 (AA)	64.40	64.60	64.80	64.60	64.60	64.60	64.80	64.40	64.60	64.60
LAB- 1 (TITR)	65.18	65.14	65.20	65.16	65.16	64.88	64.84	65.07	64.82	64.89
LAB- 1 (TITR)	64.68	64.67	64.57	64.58	64.32	64.37	64.52	64.58	64.58	64.55
LAB- 5 (TITR)	65.19	65.12	65.18	65.18	65.12	64.82	64.86	64.90	64.80	64.93
LAB- 6 (TITR)	64.82	64.42	64.27	64.37	64.45	64.21	64.56	64.16	64.49	64.63
LAB- 9 (TITR)	65.00	65.00	65.20	65.00	65.20	65.00	64.60	65.00	65.20	65.20
LAB-13 (TITR)	65.07	65.07	64.97	64.97	64.97	65.07	64.97	65.07	64.97	64.97
LAB-14 (TITR)	65.27	65.27	65.07	65.17	65.17	65.17	65.17	65.17	65.07	65.17
LAB-16 (TITR)	64.78	64.60	64.71	64.64	64.58	64.56	64.58	64.60	64.66	64.59
LAB-18 (TITR)	64.83	64.96	65.03	64.89	64.96	64.95	64.86	64.96	64.96	64.96
	64.78	64.85								
LAB-21 (TITR)	64.91	64.85	64.96	64.88	64.92	64.92	64.91	64.87	64.98	64.88
LAB-23 (TITR)	64.80	64.80	64.70	64.60	64.40	64.60	64.60	64.70	64.70	64.60
LAB-26 (TITR)	64.45	64.32	64.16	63.90	64.10	64.02	63.93	64.01	64.12	63.92
LAB-30 (TITR)	65.06	64.86	65.06	64.86	64.86	64.86	64.86	64.86	64.86	64.96
LAB-32 (TITR)	64.83	64.62	64.57	64.52	64.52	64.42	64.62	64.62	64.57	64.62
LAB-34 (TITR)	65.02	65.14	65.04	64.74	64.95	64.72	64.86	64.82	64.80	64.66
LAB-35 (TITR)	64.76	64.85	64.65	64.66	64.69	64.78	64.85	64.97	64.77	64.78
	64.81	64.89								
LAB-37 (TITR)	64.59	64.70	64.60	64.48	64.69	64.61	64.59	64.71	64.61	64.58
LAB-40 (TITR)	65.00	64.80	64.70	65.00	65.00	64.90	64.60	64.70	64.40	65.00
	64.90	64.80	64.80	64.70	64.80	64.70	64.70	64.40	64.90	
LAB-45 (TITR)	64.68	64.67	64.62	64.68	64.68	64.67	64.60	64.65	64.59	64.62
LAB-24 (GRAV)	65.31	65.49	65.42	65.51	65.37	65.38	65.48	65.44	65.35	65.40
LAB-29 (GRAV)	64.26	64.24	64.26	64.24	64.24	64.24	64.19	64.21	64.26	64.29
*LAB-31 (GRAV)	63.64	63.54	63.52	63.52	64.03	63.83	63.59	63.78	63.49	
LAB-33 (GRAV)	64.56	64.53	64.52	64.58	64.44	64.59	64.53	64.55	64.55	64.47
LAB-38 (GRAV)	64.22	64.23	64.46	64.57	64.73	64.34	64.25	63.87	64.37	64.46
LAB-39 (GRAV)	63.98	64.24	64.16	64.34	64.28					
*LAB-39 (GRAV)	62.02	62.11	62.02	61.76	61.38					

S (wt %)

LAB- 5 (GRAV)	17.98	18.04	18.09	18.10	18.10	18.01	17.98	17.96	17.96	18.01
LAB- 6 (GRAV)	17.52	17.58	17.47	17.56	17.54	17.60	17.57	17.68	17.72	17.70
LAB- 9 (GRAV)	17.57	17.60	17.60	17.53	17.51	17.56	17.60	17.60	17.53	17.57
LAB-13 (GRAV)	17.90	17.94	18.01	17.97	17.99	17.91	17.83	17.95	17.93	18.01
LAB-14 (GRAV)	17.95	17.91	17.82	17.94	17.99	17.83	17.78	17.86	18.04	17.92
LAB-21 (GRAV)	18.12	18.02	18.03	17.82	17.88	18.00	18.09	17.91	18.19	18.00
LAB-23 (GRAV)	17.66	17.83	17.86	17.80	17.91	17.70	17.68	17.74	17.65	17.70
*LAB-30 (GRAV)	16.60	16.90	17.30	17.20	16.60	16.80	16.70	16.50	16.80	17.00
LAB-31 (GRAV)	17.54	17.49	17.78	17.51	17.72	17.69	17.73	17.63	17.89	17.65
LAB-34 (GRAV)	17.65	17.64	17.65	17.61	17.71	17.64	17.76	17.80	17.60	17.60
LAB-35 (GRAV)	17.77	18.06	17.79	17.84						
LAB-38 (GRAV)	18.05	18.08	18.00	17.97	18.07	18.07	18.07	18.00	18.03	
LAB-39 (GRAV)	17.98	18.02	18.10	18.06	18.10					
*LAB-39 (GRAV)	19.14	18.66	18.68	18.92	18.88					
LAB-14 (COMB)	17.35	17.75	17.50	17.75	17.90	17.60	17.60	17.75	17.65	17.65
LAB-18 (COMB)	17.85	17.92	17.77	17.81	17.85	17.90	17.90	17.90		
LAB-26 (COMB)	18.40	18.20	18.20	18.00	18.40	18.20	18.30	18.40	18.00	18.40

Sb (wt %)

LAB- 5 (AA)	.360	.360	.370	.370	.370	.360	.370	.370	.370	.360
LAB- 6 (AA)	.399	.404	.394	.397	.408	.400	.397	.400	.406	.395
LAB- 9 (AA)	.332	.326	.344	.314	.324	.326	.336	.334	.332	.364
LAB-14 (AA)	.371	.367	.376	.367	.360	.371	.363	.365	.366	.365
*LAB-19 (AA)	.299	.295	.300	.296	.297	.300	.299	.298	.298	.299
LAB-21 (AA)	.343	.343	.342	.342	.342	.348	.347	.349	.348	.347
LAB-24 (AA)	.366	.374	.372	.374	.362	.372	.378	.374	.377	.374
LAB-30 (AA)	.360	.350	.350	.360	.360	.360	.360	.360	.360	.360
LAB-34 (AA)	.336	.331	.321	.331	.336	.329	.328	.335	.333	.333
LAB-35 (AA)	.380	.390	.380	.390						
LAB- 3 (COLOR)	.376	.366	.376	.369	.367	.376	.371	.366	.364	.365
LAB-23 (COLOR)	.364	.324	.364	.344	.348	.328	.364	.360	.372	.348
LAB-39 (ES)	.330	.340	.314	.318	.325					
LAB-40 (ES)	.300	.320	.330	.340	.360	.300	.320	.320	.350	.330

TABLE 8 (cont'd)

Se (µg/g)

LAB-18 (AA)	35	34	33	33	32	34				
LAB-24 (AA)	31	35	29	29	29	35	32	36	29	36
LAB-34 (AA)	30	30	29	29	29	29	30	29	30	29
LAB- 1 (COLOR)	30	30	31	31	32	33	30	31	30	29
LAB- 3 (COLOR)	33	32	32	33	32	33	32	32	33	33
LAB- 9 (COLOR)	27	24	27	25	22	27	24	27	25	26
LAB-23 (COLOR)	35	36	36	37	36	36	37	36	35	36

SiO_2 (wt %)

LAB- 2 (AA)	.73	.73	.73	.72	.73	.72	.72	.72	.74	.73
LAB-26 (AA)	.82	.71	.71	.78	.71	.82	.71	.82	.71	.78
LAB- 1 (GRAV)	.73	.73	.73	.75	.71	.68	.68	.68	.68	.68
LAB- 4 (GRAV)	.73	.72	.72	.72	.71	.72	.71	.71	.72	.71
LAB- 5 (GRAV)	.64	.64	.63	.67	.66	.63	.64	.65	.62	.63
*LAB- 9 (GRAV)	1.15	1.05	1.05	1.05	1.15	1.05	1.15	1.15	1.05	1.15
LAB-13 (GRAV)	.78	.73	.78	.71	.78	.81	.80	.83	.80	.79
LAB-14 (GRAV)	.77	.81	.82	.81	.81	.86	.83	.89	.98	.78
*LAB-18 (GRAV)	.53	.57	.53	.55	.62	.60	.55	.60		
LAB-19 (GRAV)	.77	.69	.72	.78	.77	.78	.81	.75	.75	.72
*LAB-21 (GRAV)	.60	.60	.60	.60	.59	.58	.59	.60	.59	.57
LAB-23 (GRAV)	.80	.81	.77	.82	.79	.82	.80	.82	.79	.80
LAB-24 (GRAV)	.70	.72	.80	.72	.80	.76	.77	.77	.73	.72
LAB-29 (GRAV)	.68	.72	.72	.72	.72	.60	.68	.68	.72	.72
LAB-34 (GRAV)	.77	.77	.74	.76	.76	.77	.76	.74	.75	.75
LAB-35 (GRAV)	.61	.65	.66	.65	.63	.63	.69	.66		
*LAB-39 (GRAV)	.30	.24	.50	.55	.60					
*LAB-39 (GRAV)	1.02	1.10	1.04	1.03	1.09					

Sn (wt %)

LAB-23 (AA)	.020	.020	.025	.020	.025	.020	.020	.020	.020	.020
	.020	.025	.025	.025	.020	.020	.020	.020	.020	.020
LAB-34 (AA)	.015	.016	.016	.016	.016	.018	.016	.016	.017	.015
LAB- 1 (COLOR)	.021	.016	.020	.019	.019	.019	.020	.019	.018	.018
*LAB- 9 (ES)	.038	.040	.043	.044	.042	.037	.042	.043	.041	.042
LAB-18 (ES)	.019	.019	.019	.019						
LAB-39 (ES)	.030	.026	.031	.027	.028					
LAB-40 (ES)	.030	.032	.030	.030	.028	.033	.033	.030	.031	.028

Te (µg/g)

LAB-34 (AA)	.5	.5	.5	.5	.5	.4	.4	.4	.4	.4
LAB-18 (COLOR)	.3	.3	.3	.3						
LAB-23 (COLOR)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0

TABLE 8 (cont'd)

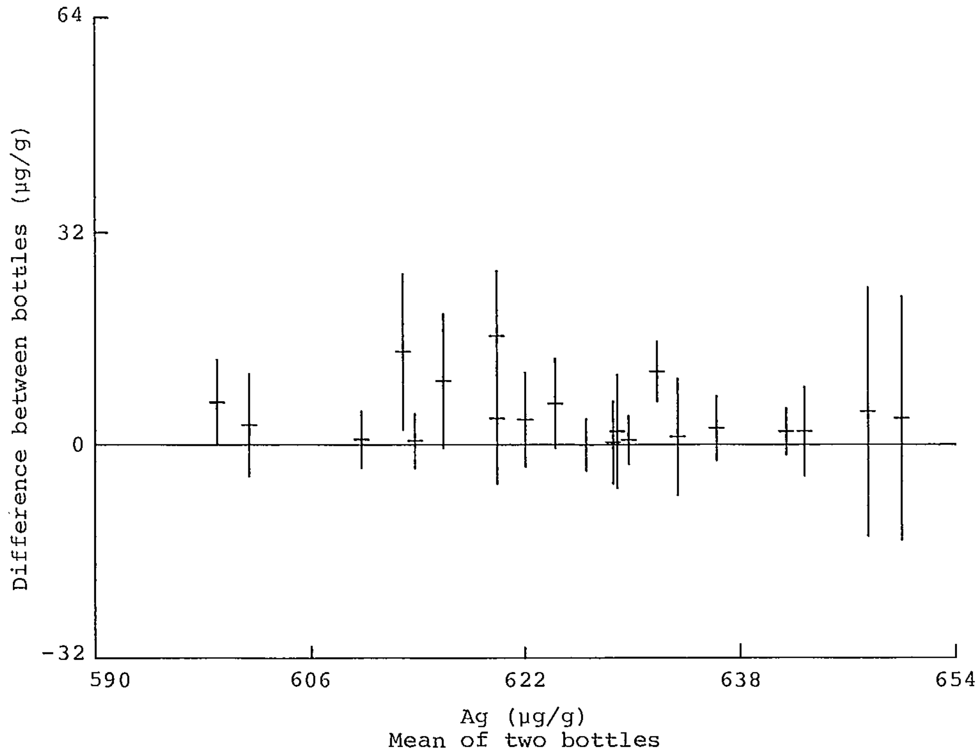
Zn (wt %)

LAB- 1 (AA)	4.45	4.45	4.46	4.47	4.42	4.47	4.46	4.46	4.40	4.42
LAB- 2 (AA)	4.38	4.45	4.43	4.43	4.45	4.45	4.44	4.43	4.46	4.45
LAB- 6 (AA)	4.44	4.41	4.38	4.41	4.42	4.37	4.41	4.41	4.41	4.40
LAB- 9 (AA)	4.35	4.29	4.29	4.25	4.28	4.27	4.28	4.29	4.25	4.28
LAB-16 (AA)	4.35	4.30	4.29	4.29	4.30	4.26	4.28	4.26	4.32	4.33
LAB-19 (AA)	4.47	4.53	4.50	4.50	4.47	4.50	4.48	4.48	4.50	4.52
LAB-21 (AA)	4.32	4.30	4.33	4.33	4.32	4.40	4.38	4.38	4.38	4.43
*LAB-23 (AA)	4.50	4.60	4.60	4.50	4.50	4.50	4.60	4.50	4.50	4.50
LAB-24 (AA)	4.44	4.45	4.46	4.48	4.50	4.50	4.52	4.56	4.49	4.50
LAB-26 (AA)	4.42	4.44	4.36	4.52	4.46	4.40	4.42	4.32	4.54	4.44
LAB-31 (AA)	4.32	4.34	4.32	4.37	4.32	4.33	4.41	4.36	4.31	4.34
*LAB-32 (AA)	5.05	5.10	5.05	5.05	5.05	5.05	5.00	5.00	5.15	5.05
LAB-34 (AA)	4.44	4.48	4.95*	4.54	4.36	4.43	4.44	4.35	4.35	4.42
LAB-35 (AA)	4.42	4.49	4.31	4.32	4.43	4.24	4.44	4.33		
LAB-38 (AA)	4.34	4.34	4.33	4.27	4.38	4.33	4.24	4.35	4.21	4.31
LAB-39 (AA)	4.28	4.40	4.16	4.20	4.24					
LAB-40 (AA)	4.10	4.38	4.35	4.38	4.40	4.35	4.10	4.30	4.35	4.38
	4.40	4.30								
LAB- 4 (TITR)	4.49	4.46	4.49	4.45	4.44	4.46	4.47	4.45	4.43	4.42
LAB- 5 (TITR)	4.30	4.36	4.32	4.30	4.30	4.24	4.24	4.24	4.14*	4.24
LAB-13 (TITR)	4.43	4.47	4.47	4.43	4.46	4.43	4.44	4.47	4.43	4.43
LAB-14 (TITR)	4.56	4.56	4.56	4.56	4.56	4.56	4.56	4.53	4.56	4.53
LAB-18 (TITR)	4.59	4.65	4.68	4.68	4.69	4.63	4.56	4.72	4.72	4.65
	4.68									
LAB-24 (TITR)	4.55	4.60	4.57	4.46	4.62	4.61	4.58	4.63	4.62	4.52
LAB-30 (TITR)	4.43	4.43	4.41	4.41	4.43	4.38	4.41	4.41	4.42	4.41
LAB-33 (TITR)	4.32	4.34	4.32	4.28	4.46	4.44				
LAB-33 (TITR)	4.46	4.55	4.47	4.47						
LAB-39 (TITR)	4.58	4.58	4.27	4.37	4.32					
LAB-45 (TITR)	4.45	4.45	4.44	4.45	4.44	4.45	4.44	4.45	4.45	4.46
LAB-45 (TITR)	4.41	4.42	4.41	4.41	4.40	4.44	4.42	4.42	4.43	4.42

* Outliers, not used for computations.

NOTE: Results are expressed on a dry basis; some have been rounded off for presentation.

LEGEND: AA - atomic absorption; TITR - titrimetry; COLOR - colorimetry (spectrophotometry); GRAV - gravimetry; ES - emission spectroscopy; FA = fire assay with gravimetric finish; FA-AA - fire assay with atomic absorption finish; NAA - neutron activation analysis; COMB - combustion.



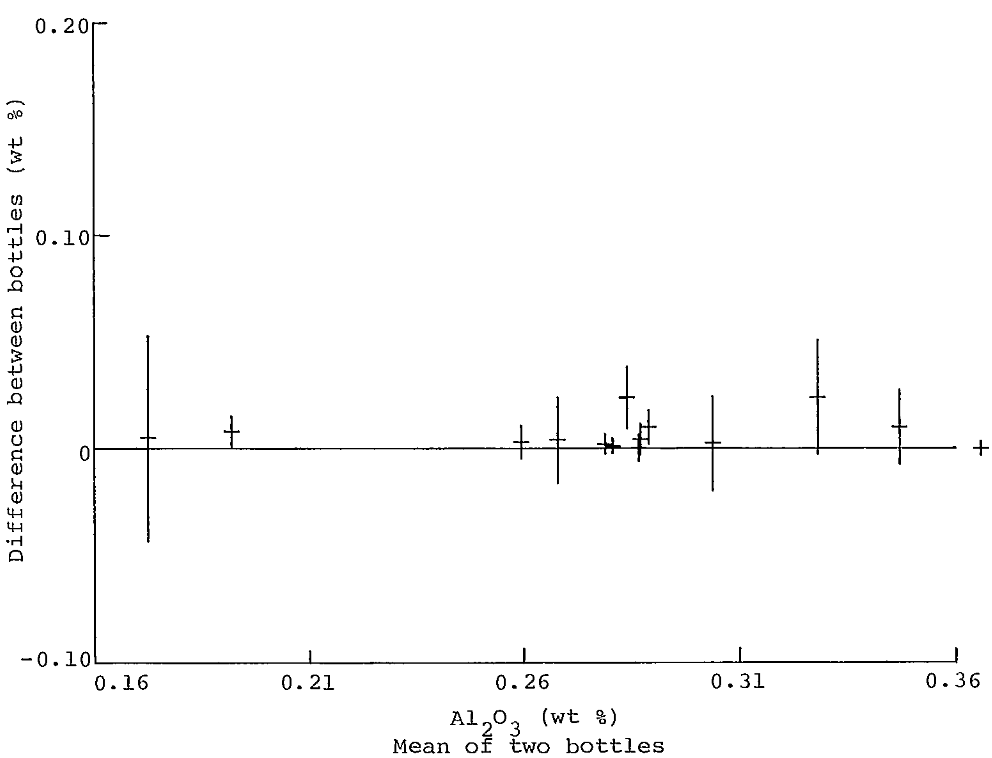
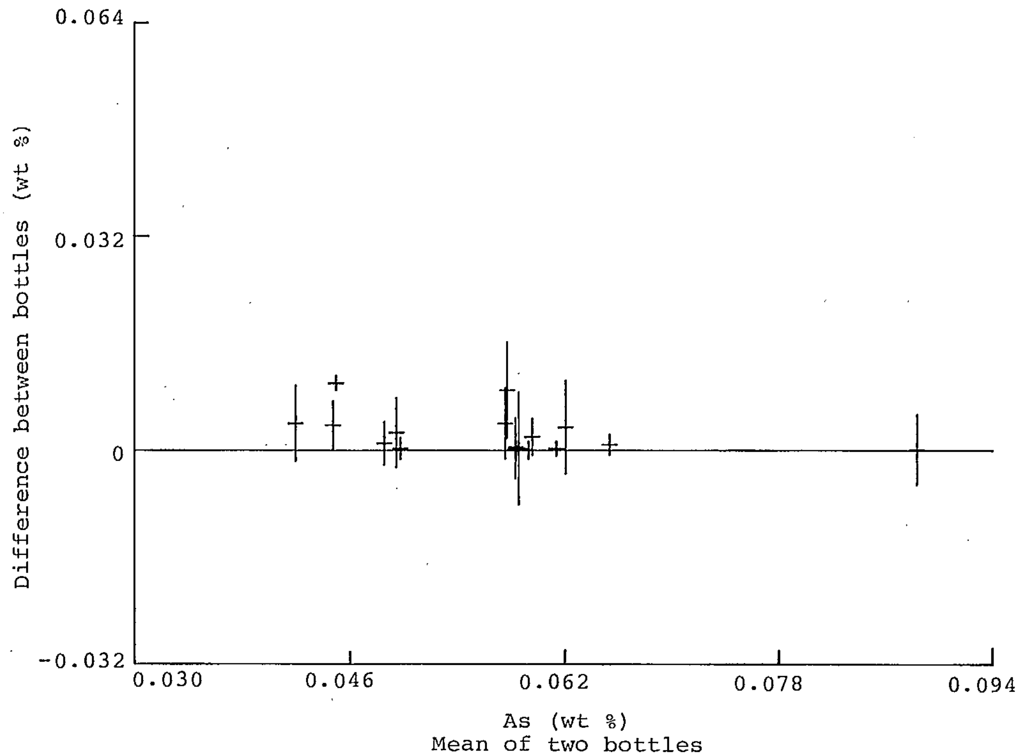


Fig. 1 - Degree of homogeneity of CPB-1. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.


0.064
0.032
0
-0.032
Difference between bottles (wt %)
0.030
0.046
0.062
0.078
0.094
As (wt %)
Mean of two bottles

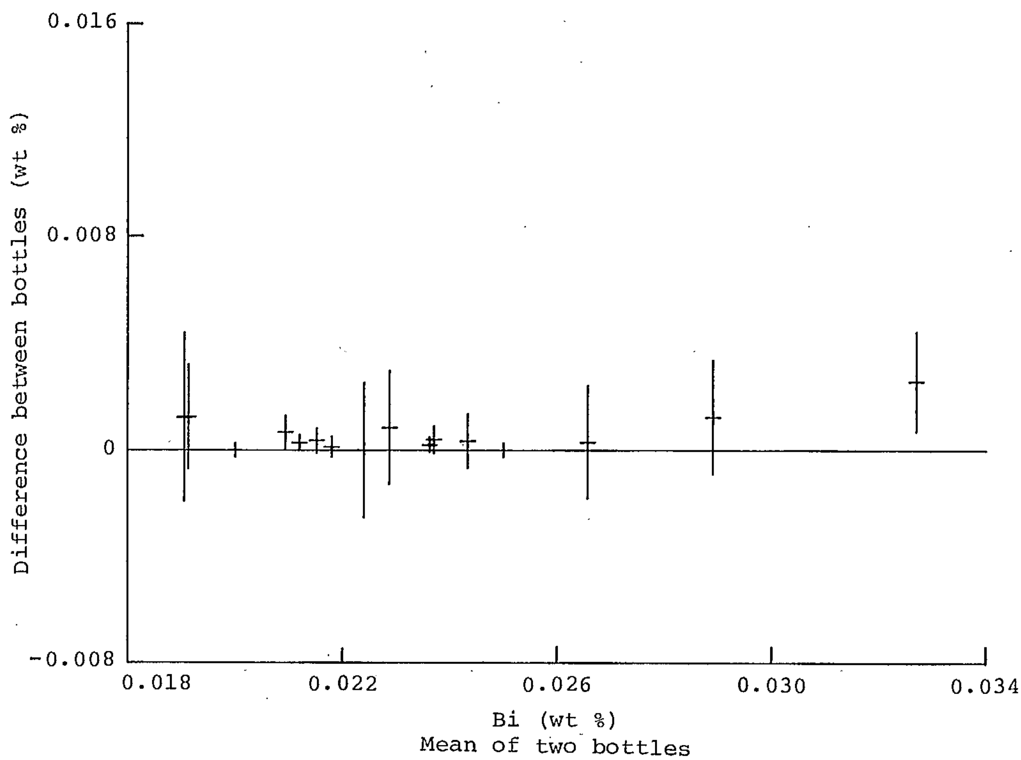
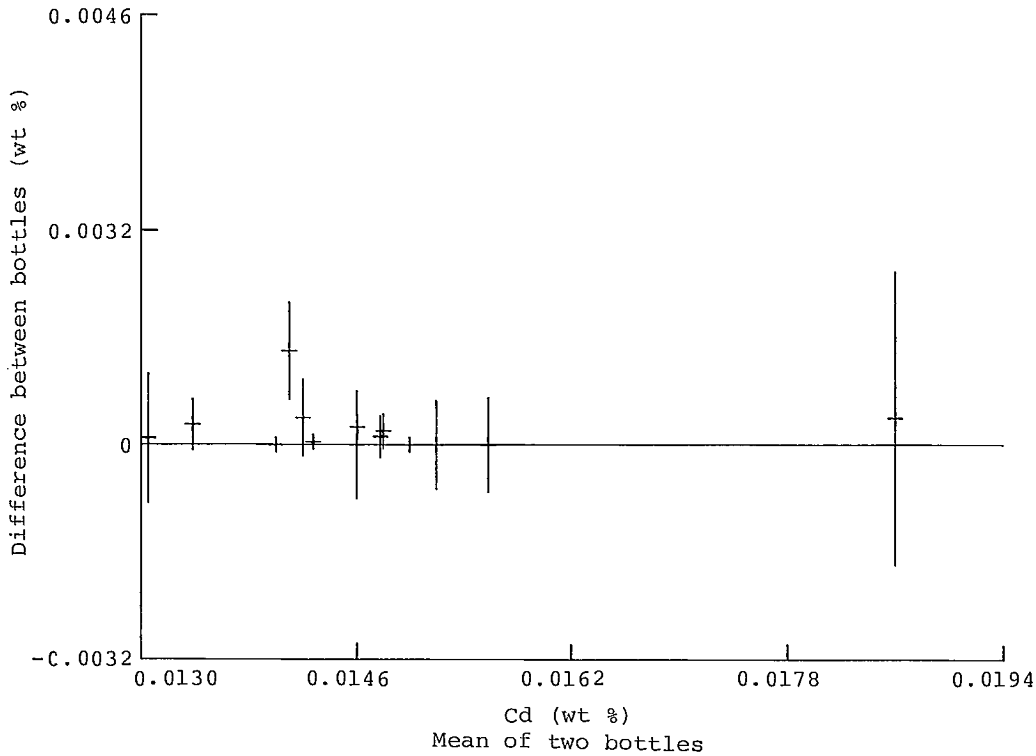
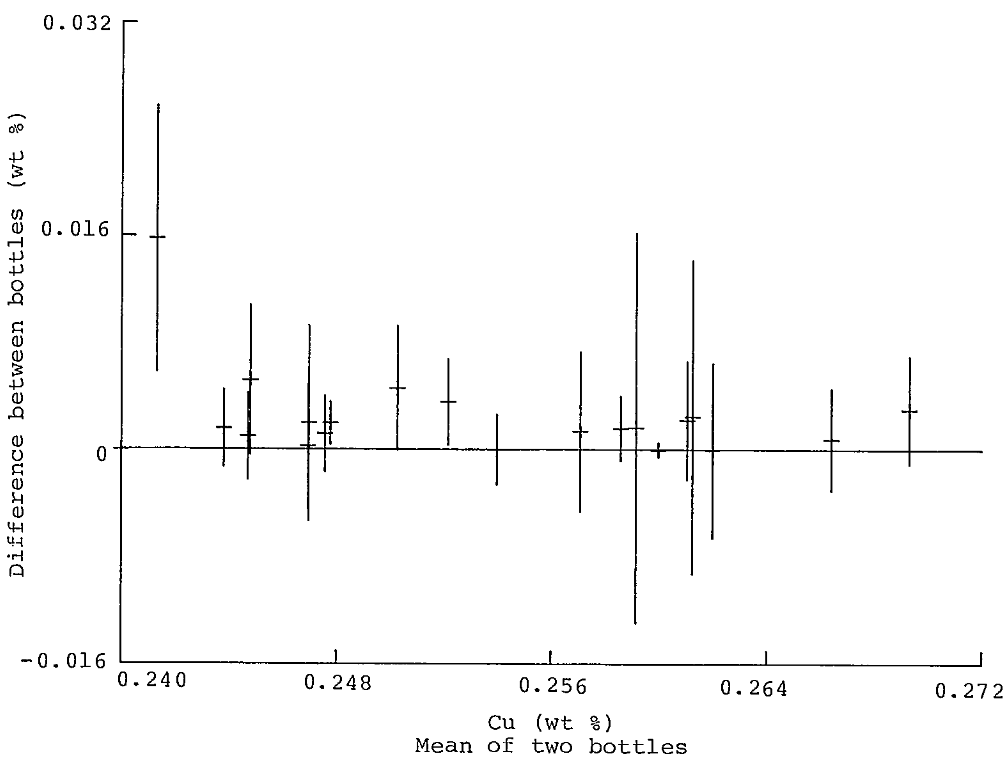

0.016
0.008
0
-0.008
Difference between bottles (wt %)
0.018
0.022
0.026
0.030
0.034
Bi (wt %)
Mean of two bottles

Fig. 1 (cont'd)

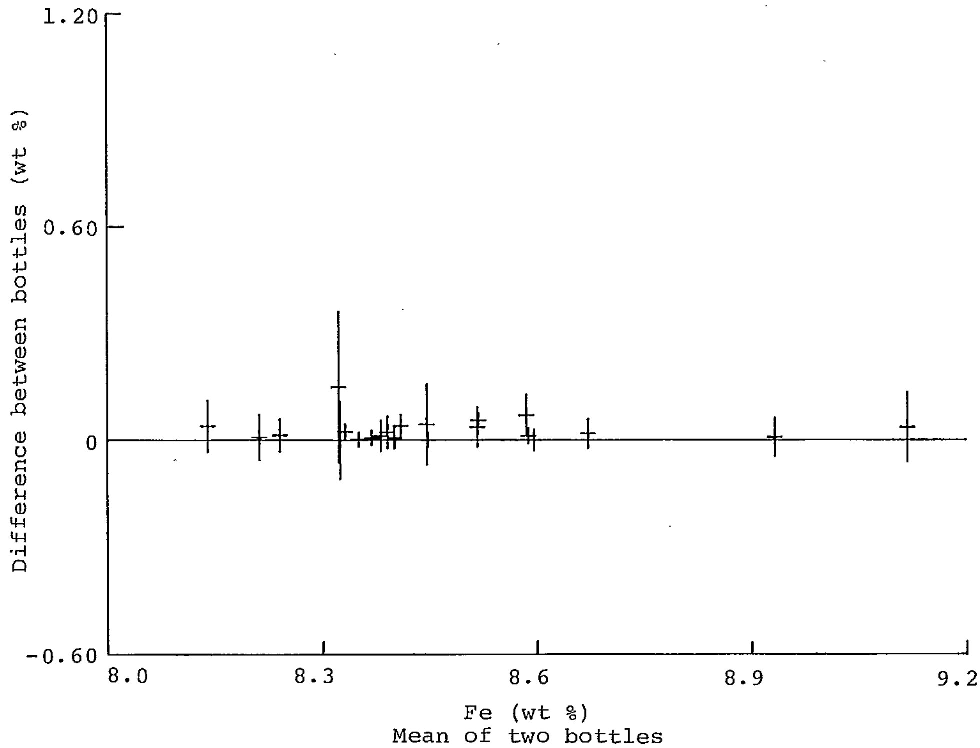


0.0046
0.0032
0
-C.0032
Difference between bottles (wt %)
0.0130
0.0146
0.0162
0.0178
0.0194
Cd (wt %)
Mean of two bottles



0.032
0.016
0
-0.016
Difference between bottles (wt %)
0.240
0.248
0.256
0.264
0.272
Cu (wt %)
Mean of two bottles

Fig. 1 (cont'd)


1.20
0.60
0
-0.60
Difference between bottles (wt %)
8.0
8.3
8.6
8.9
9.2
Fe (wt %)
Mean of two bottles

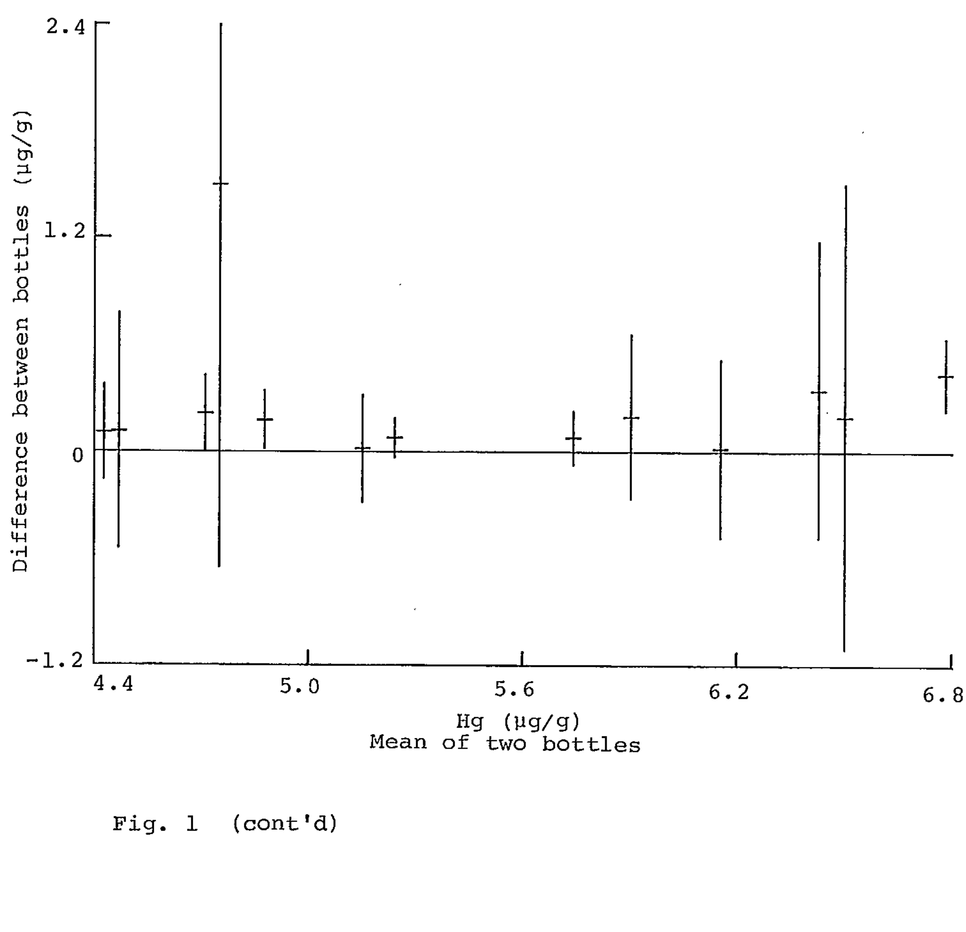
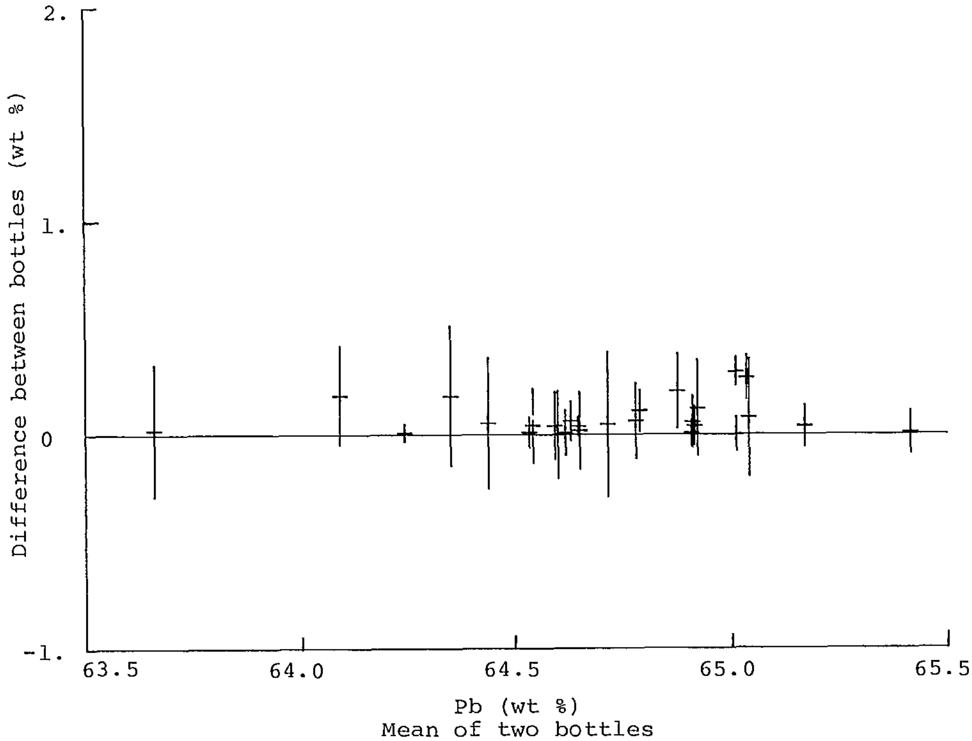

2.4
1.2
0
-1.2
Difference between bottles (µg/g)
4.4
5.0
5.6
6.2
6.8
Hg (µg/g)
Mean of two bottles

Fig. 1 (cont'd)


2.
1.
0
-1.
Difference between bottles (wt %)
63.5
64.0
64.5
65.0
65.5
Pb (wt %)
Mean of two bottles

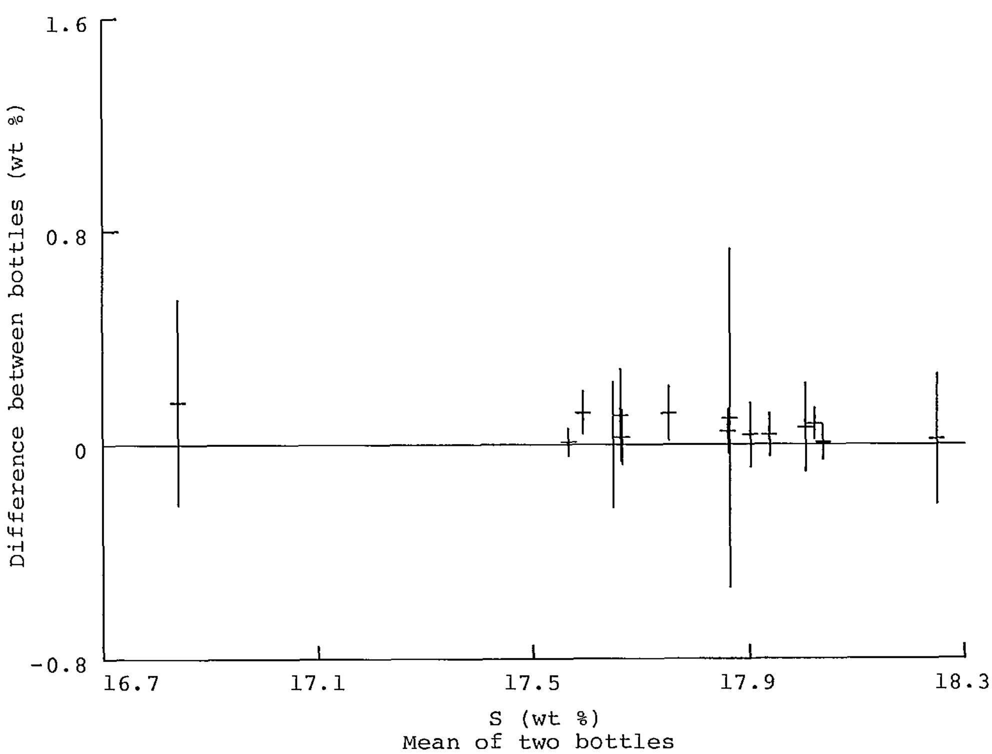
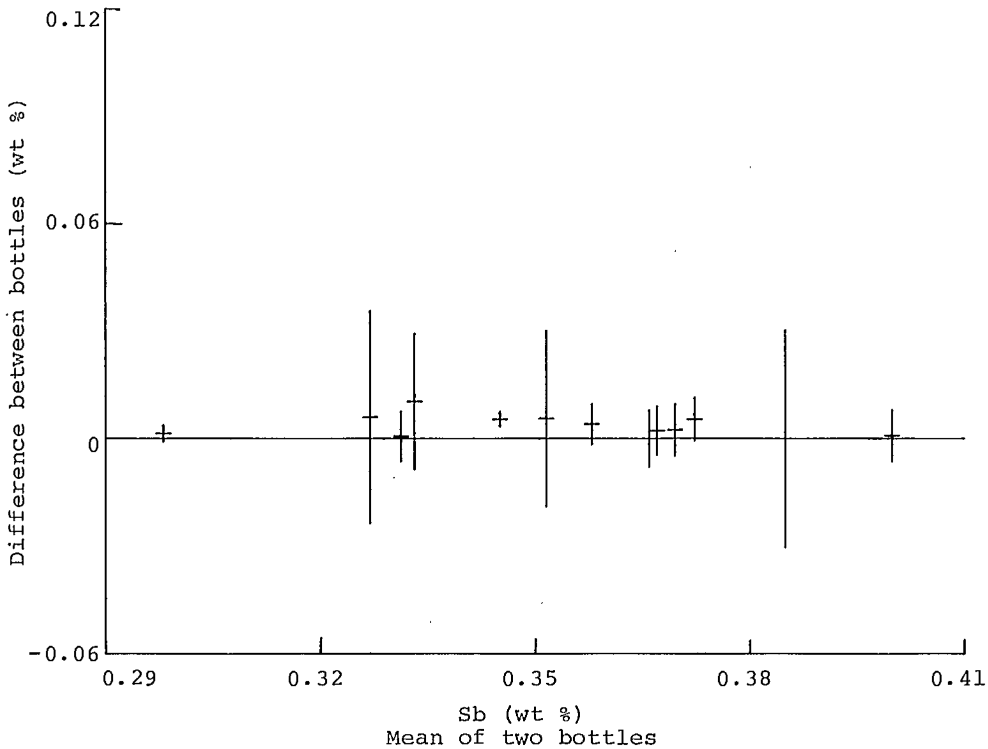

1.6
0.8
0
-0.8
Difference between bottles (wt %)
16.7
17.1
17.5
17.9
18.3
S (wt %)
Mean of two bottles

Fig. 1 (cont'd)


0.12
0.06
0
-0.06
Difference between bottles (wt %)
0.29
0.32
0.35
0.38
0.41
Sb (wt %)
Mean of two bottles

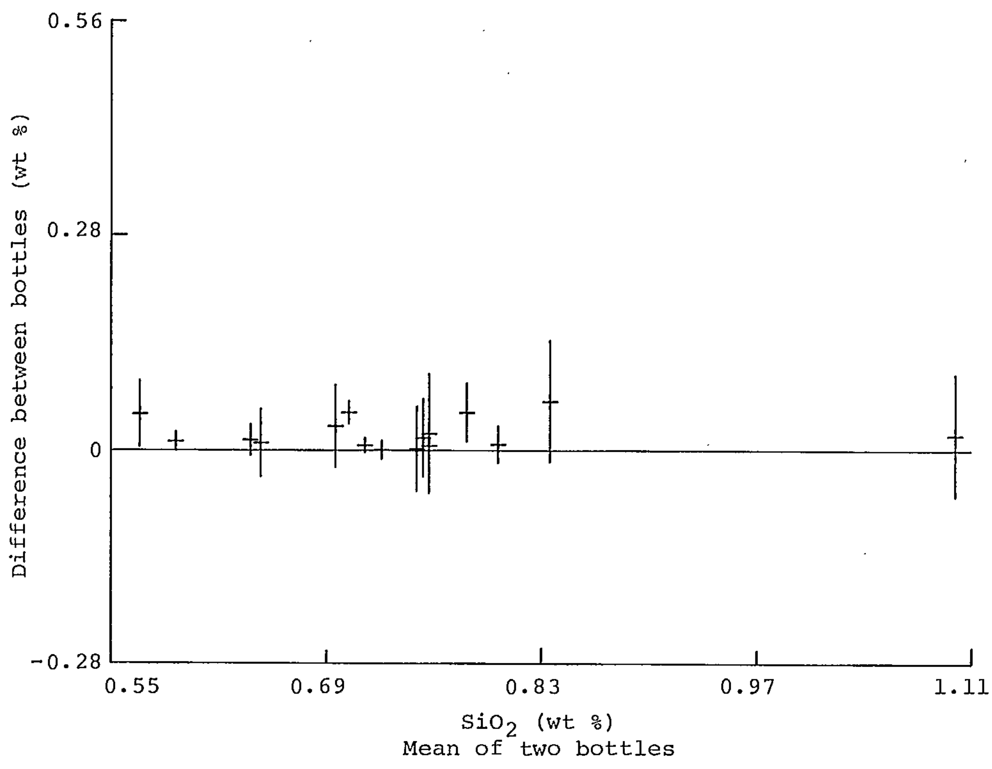

0.56
0.28
0
-0.28
Difference between bottles (wt %)
0.55
0.69
0.83
0.97
1.11
SiO2 (wt %)
Mean of two bottles

Fig. 1 (cont'd)

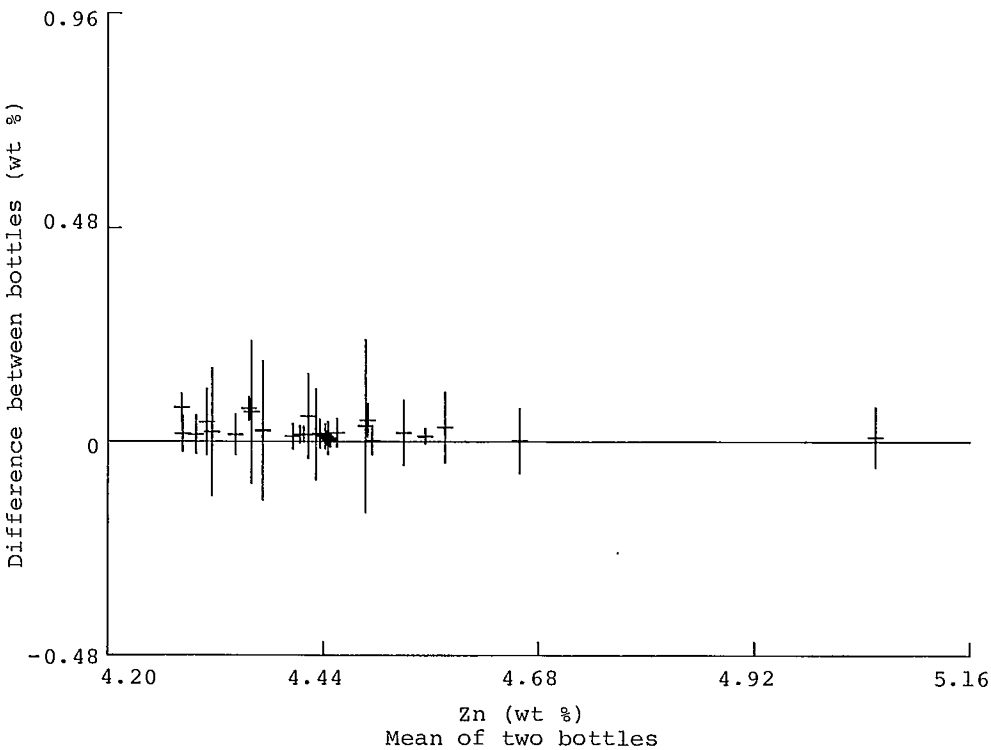

0.96
0.48
0
-0.48
Difference between bottles (wt %)
4.20
4.44
4.68
4.92
5.16
Zn (wt %)
Mean of two bottles

Fig. 1 (cont'd)

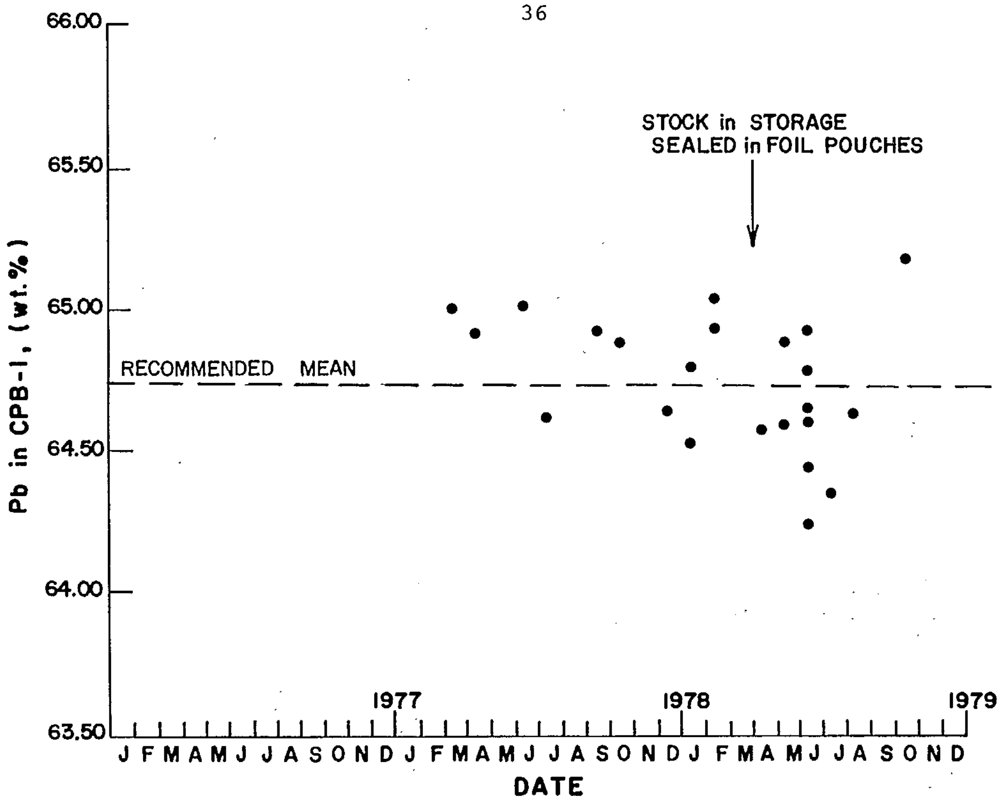


Fig. 2 Laboratory means for Pb vs date of analysis

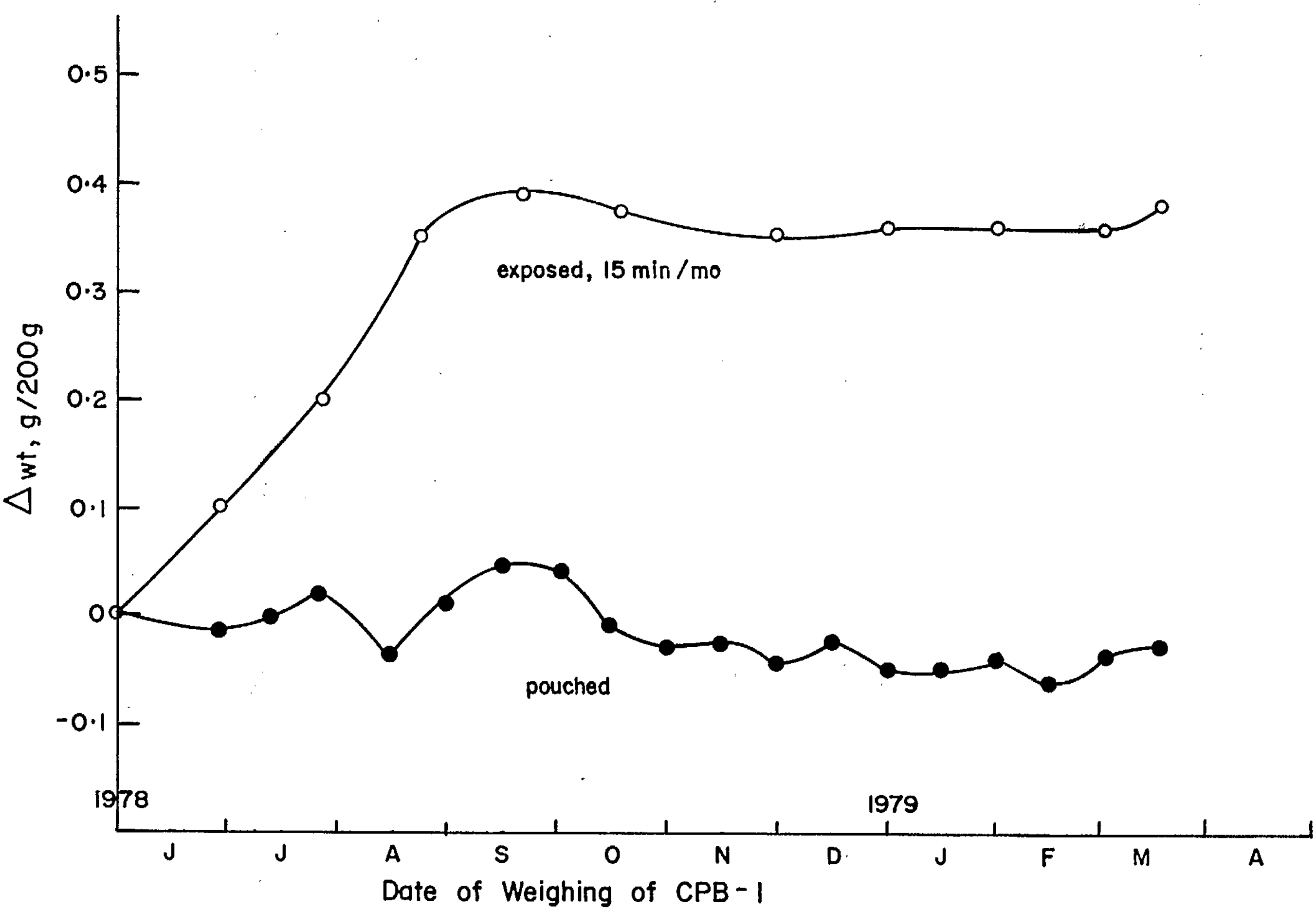


Fig. 3 Change in weight of pouched and unpouched samples of CPB-1 vs storage time

APPENDIX A

PARTICIPATING LABORATORIES

Alfred H. Knight Ltd., Wallasey, Cheshire, England.

Bondar-Clegg and Company Ltd., Ottawa, Ontario.

Bondar-Clegg and Company Ltd., North Vancouver, British Columbia.

Britannia Lead Company Ltd., Gravesend, Kent, England.

Canada Centre for Mineral and Energy Technology, Mineral Sciences Laboratories, Department of Energy, Mines and Resources, Ottawa, Ontario (six independent analysts).

Chemex Labs Ltd., North Vancouver, British Columbia.

Cominco Ltd., Trail, British Columbia.

Commonwealth Smelting Ltd., Avonmouth, Bristol, England.

Falconbridge Copper Ltd., Lake Dufault Division, Noranda, Quebec.

General Testing Laboratories, Vancouver, British Columbia.

Geological Survey of India, Central Chemical Laboratory, Calcutta, India (two independent analysts).

Geological Survey of Norway, Trondheim, Norway.

Geological Survey of West Malaysia, Ipoh, Perak, Malaysia.

Hudson Bay Mining and Smelting Company Ltd., Flin Flon, Manitoba.

Inco Ltd., Analytical Services, Process Technology, Copper Cliff, Ontario.

Irish Base Metals, Tynagh, Galway, Ireland.

Ledoux and Company, Teaneck, New Jersey, U.S.A.

LKAB Prospektering AB, Geochemical Laboratory, Stockholm, Sweden.

Loring Laboratories Ltd., Calgary, Alberta.

Newmont Exploration Limited, Danbury, Connecticut, U.S.A.

National Institute for Metallurgy, Randburg, South Africa.

Noranda Research Centre, Pointe Claire, Quebec.

Ontario Ministry of Natural Resources, Mineral Research Branch, Toronto, Ontario.

Sulphide Corporation Pty. Ltd., Boolaroo, N.S.W., Australia.

The Broken Hill Associated Smelters Proprietary Ltd., Port Pirie, South Australia.

APPENDIX B

OUTLINE OF PRINCIPAL TITRIMETRIC METHODS USED FOR LEAD IN CPB-1

The titrimetric methods for lead outlined below were used by a relatively large proportion of contributing laboratories and they can not be conveniently summarized in Table 5. It is possible that the procedures of individual laboratories may have differed in some minor details from the outlines given; however, it is unlikely that this would be of significance in the correlation of methods and means.

Molybdate method (Laboratories 6, 9, 13, 21, 34)

After sample decomposition and fuming with sulphuric acid, lead was separated as the sulphate. The precipitate was dissolved by boiling in ammonium acetate-acetic acid solution and, while hot, the solution was titrated with ammonium molybdate solution using tannic acid as an external indicator.

EDTA method (Laboratories 1, 1a, 5, 37)

After sample decomposition and fuming with sulphuric acid, lead was separated as the sulphate by filtration. Lead remaining in the filtrate was determined by atomic-absorption spectrophotometry in the case of Labs 1 and 1a. The lead sulphate was dissolved either in hydrochloric acid-sodium chloride solution (Labs 1 and 1a), or in acetate solution. After pH adjustment the solution was titrated with EDTA either at room temperature or just below the boiling point (Lab 37).