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ZINC CONCENTRATE CZN-1 — A CERTIFIED REFERENCE MATERIAL

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

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ZINC CONCENTRATE CZN-1 — A CERTIFIED REFERENCE MATERIAL

by

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

SYNOPSIS

A 300-kg sample of a zinc (flotation) concentrate, CZN-1, from Kimberley, British Columbia, has been prepared as a compositional reference material. CZN-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, 30 laboratories contributed results for one or more of 21 elements in each of two bottles of CZN-1. Based on a statistical analysis of the data, recommended values have been assigned for: Ag, Al_2O_3, As, Cd, Cu, Fe, Hg, Mn, Pb, S, Sb and Zn. Also, non-certified values have been determined for: Au, Bi, CaO, In, MgO, Se, SiO_2 and Sn.

* Research Scientist, **Technologist, and ***Research Scientist, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.

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 ZINC CZN-1 — UN MATERIAU DE REFERENCE CERTIFIE

par

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

SOMMAIRE

Un échantillon de 300 kg de concentré de zinc (flottation), CZN-1, provenant de Kimberley en Colombie-Britannique, a été préparé en tant que matériau de référence de composition. Le CZN-1 a été broyé 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.

Trente laboratoires ont fourné des résultats pour un ou plusieurs des 21 éléments dans chacune des deux bouteilles de CZN-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, Cd, Cu, Fe, Hg, Mn, Pb, S, Sb et Zn. Aussi des valeurs non-homologuées ont été déterminées pour l'Au, Bi, CaO, In, MgO, Se, SiO_2 et Sn.

*Chercheur scientifique, **Technologue, et ***Chercheur scientifique, Laboratoires des sciences minérales, CANMET, Energie, Mines et Ressources Canada, Ottawa.

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 zinc concentrate, CZN-1, for use as a 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).

CZN-1 was chosen as a reference material because of its mineralogical complexity and because it contains a relatively large number of minor and trace elements at analytically 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 30 laboratories which used methods of their choice. Recommended values for the 12 certified elements are given in Table 3; statistical, methodological and other analytical information is presented in Tables 4 to 8.

PREPARATION OF CZN-1

In early 1976, CZN-1, which had been dried at ~100°C, was ground to pass a 74-µm screen. The powdered concentrate, weighing approximately 300 kg, was tumbled in a 570-L conical blender for 10 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 CZN-1

Mineral	wt %
Sphalerite (~ 10% Fe)	84
Galena	8.5
Pyrrhotite	4
Pyrite	1
Iron oxides	1
Quartz	0.5
Aluminosilicates	0.5
Carbonates	0.5

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

TABLE 2

Particle size analysis of CZN-1 (wet screen)

Size of fraction (µm)	wt %
-74 +55	3
-55 +46	4.5
-46 +37	3.5
-37	89

INTERLABORATORY PROGRAM FOR CERTIFICATION OF CZN-1

The laboratories that participated in the certification program for CZN-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 (Tables 5, 6) 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 were subsequently corrected for the 0.2% moisture content of CZN-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 12 constituents certified, the rejection rate was 11%. This is typical of previous CCRMP ore certification programs and is considered acceptable. The degree of homogeneity of CZN-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 jth 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:

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

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	$\sum_{i}^{k}\sum_{j}^{n_i} (x_{ij}-\bar{x}_{..})^2$	$\sum_{i}^{k} n_i - 1$		

where:

n_i = the number of results reported in set i;

k = the number of sets.

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:

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

where 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 certification factors for the 12 certified constituents of CZN-1 are given in Table 3 along with the consensus values which are boxed in for easy identification.

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

Discussion of analytical results

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

Table 5 gives a methodological classification of results 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 subsample decomposition or 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

Most results accepted for alumina, lime and magnesia were obtained by atomic absorption on a sinlge solution prepared from a separate subsample of CZN-1. The solution

was also used for determining other elements in some cases. 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 from solutions to which La or Sr had been added as a buffer or releasing agent. As expected, significant differences were not found among results by the two types of decomposition or by the conditioning of the sample solution.

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 all but 3 of 14 such 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 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 CF.

Copper and cadmium

With four exceptions all results for copper and cadmium 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 for copper and cadmium 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

Of the 15 accepted sets of results for mercury, 13 were by the atomic absorption cold-vapour technique (4). Most analysts used a mixture of nitric and hydrochloric acids in a low-temperature decomposition; some also used a second oxidizing agent such as $KMnO_4$, $KClO_3$ or Br_2. Stannous chloride was most commonly used as reductant in the cold-vapour generator.

It should be noted that, with the exception of Labs 5 and 13, all laboratories analyzed subsamples of CZN-1 that had been dried previously at 105°C for two hours. Because the mean value of 40 µg/g for the two sets obtained on undried material is somewhat lower than the recommended value, it is probable that the loss of mercury during the drying operation is insignificant.

Sulphur

All 14 of the accepted sets of results for sulphur were by the classical gravimetric method involving the weighing of $BaSO_4$. Thirteen laboratories used acid decomposition involving nitric acid and bromine, and one used peroxide fusion. 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 $BaSO_4$.

Two sets of results by the combustion method were rejected at the outset because this method is not considered appropriate for certifying reference material such as CZN-1.

Stability of CZN-1

Figure 2 shows the laboratory means for zinc in CZN-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 CZN-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 CZN-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 CZN-1 given in Table 3. This conclusion is supported by the data plotted in Fig. 3 which show that there was no net change in weight of protected test bottles from June 1, 1978 to March 15, 1979. Monitoring will continue throughout the life of the stock of CZN-1.

Figure 3 also shows that unprotected test bottles of CZN-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 CZN-1 under a dry inert gas in a dessicator 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.

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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	13	139	0.25	0.24	0.26	10.9	3.0	3.6
As	21	194	0.026	0.024	0.028	16.3	5.6	2.9
Cd	22	227	0.132	0.130	0.134	3.0	1.5	2.0
Cu	27	277	0.144	0.141	0.147	3.9	1.8	2.2
Fe	27	258	10.93	10.88	10.99	1.1	0.4	2.6
Mn	20	200	0.219	0.213	0.226	5.7	1.7	3.4
Pb	31	307	7.45	7.40	7.49	1.2	0.8	1.6
S	15	139	30.2	30.0	30.4	1.5	0.4	3.7
Sb	14	142	0.052	0.050	0.055	9.1	3.5	2.6
Zn	36	362	44.74	44.64	44.85	0.5	0.3	1.8
			(µg/g)	(µg/g)	(µg/g)			
Ag	22	212	93	90	95	5.0	1.9	2.6
Hg	15	144	43	40	46	15.8	5.2	3.1

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	13	134	0.25	0.23	0.27	16	3	5
MgO	14	150	0.32	0.30	0.33	10	1.6	6
SiO_2	20	186	1.0	0.9	1.1	16	3	5
			(µg/g)	(µg/g)	(µg/g)			
Au	5	59	0.09	0.03	0.14	126	10	13
Bi	14	143	32	23	40	56	10	6
In	9	77	86	69	104	41	8	5
Se	9	88	5	4	6	33	9	4*
Sn	7	68	74	52	96	60	7	9

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

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

TABLE 5(a)

Summary of analytical methods for Ag in CZN-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	94
Fire assay-atomic abs.	Pb button scorified to ∿ 2 g; dissolved in HNO_3 for a.a.	1	1	10	93
	Pb cupelled to bead; dissolved in HNO_3 for a.a.	1	9	10	84
	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	93
	Pb button partially cupelled; dissolved in HNO_3 for a.a.; Ag loss det'd with Ag-110	1	12	10	93
	Pb button cupelled to 100 mg; dissolved in HNO_3 for a.a.	1	16	10	90
Atomic absorption	HNO_3 + HCl (some with HF and/or Br_2); final soln 20-50% v/v HCl	5	10,24,30, 31,40	50	96
	HNO_3 + Br_2 + HF + H_2SO_4; fumed and residue treated with tartaric and HNO_3 for a.a.	1	15	10	94
	HNO_3 + Br_2; complex with Hg	1	35	4	96
	HNO_3 + Br_2; final soln 5% v/v HCl	1	16	10	82
	HNO_3 + HF; final soln ∿ 6% v/v HNO_3	1	26	10	82
	No details given	1	19	10	99

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

TABLE 5(b)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Colorimetric (spectrophotometric)					
Arseno-molybdate	$HNO_3 + Br_2 + HCl + H_2SO_4$; As collected with $Fe(OH)_3$, ext'd as xanthate	1	3	10	0.028
	$HNO_3 + KClO_3 + H_2SO_4$; As collected with $Fe(OH)_3$; As distilled as Cl^-	1	15	10	0.031
	$HNO_3 + HCl + KClO_3$; As distilled as Cl^-	1	23	14	0.023
	$HNO_3 + H_2SO_4$; As distilled as Cl^-	1	34	10	0.021
	Na_2O_2 fusion; cake dissolved in HCl; As (as Cl^-) ext'd with benzene	1	24	10	0.029
	Decomposition not given; As sep'd by distillation	1	18	5	0.030
Diethyldithiocarbamate	$HNO_3 + HClO_4 + H_2SO_4$; As sep'd as AsH_3, collected in diethyldithiocarbamate medium	1	9	10	0.030
	Na_2O_2 fusion; cake dissolved in HCl; As sep'd as AsH_3, collected in diethyldithiocarbamate medium	1	5	10	0.020
Iodide	$HNO_3 + HClO_4$; iodide ext'd with cyclohexane	1	40	10	0.027
Titrimetric					
	$HNO_3 + H_2SO_4$; As distilled as halogen complex, titrated iodometrically	3	10,33,35	24	0.025
	$HNO_3 + HCl + H_2SO_4$; As collected with $Fe(OH)_3$; sep'd by distillation, titrated iodometrically	1	21	10	0.024
	Details not given	1	13	10	0.034
Atomic absorption					
	$HNO_3 + Br_2$; final soln dilute HNO_3; flameless a.a.	1	16	10	0.025
	$HNO_3 + HCl + H_2SO_4$; soln treated with 5% w/v $KMnO_4$; AsH_3 generated with sodium borohydride	1	38	10	0.029
	$HNO_3 + HCl$; final soln dilute $HNO_3 + HCl$	1	30	10	0.018
	Details not given	1	32	10	0.019
Neutron activation		1	24	10	0.027
Emission spectroscopic		1	39	5	0.033
Not specified		1	14	6	0.025

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

TABLE 5(c)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Titrimetric					
Dichromate	HNO_3 + HCl + HF + H_2SO_4 or $HClO_4$; Fe ppt'd with NH_3, redissolved in HCl, reduced with $SnCl_2$	2	1,12	20	10.84
	HNO_3 + HCl + H_2SO_4; as above	4	24,29,31,34	40	10.93
	HNO_3 + HCl + H_2SO_4; Fe ppt'd with NH_3; reduced with Ag-reductor	2	39,39a	10	10.96
	HNO_3 + HCl + H_2SO_4; Fe reduced with Al°	1	30	10	10.71
	Na_2O_2 fusion; cake dissolved in dilute HCl; Fe reduced with $SnCl_2$	4	5,6,23,35	34	11.01
	Decomposition not given; Fe reduced with H_2S	1	18	12	11.18
	Details not given	3	13,14,16a	30	10.92
Permanganate	Sample roasted, treated with HNO_3 + HCl + HF + H_2SO_4; Fe reduced with $SnCl_2$	1	26	10	10.77
Ceric amm. sulphate	Na_2O_2 + NaOH fusion; cake dissolved in HCl; Fe ppt'd with NH_3; Fe reduced with Pb°	1	9	10	10.76
Atomic absorption					
	HNO_3 + HCl + HF + $HClO_4$; Fe + Pb ppt'd with NH_3 + $(NH_4)_2CO_3$, dissolved in HCl	1	1	10	11.00
	HNO_3 + HCl + HF + $HClO_4$; final soln 1% v/v HCl and 10% v/v HNO_3	1	15	10	10.99
	HNO_3 + HCl + HF; final soln 1% v/v HNO_3 containing EDTA	1	40	12	10.74
	HNO_3 + HCl + HF; final soln 1% La and 5% v/v HCl	1	12	10	11.00
	HNO_3 + HF + $HClO_4$; other details not given	1	38	10	11.20
	HNO_3 + HBr; other details not given	1	22	10	10.93
	Mixed acid digestion + Na_2O_2 fusion of residue; final soln dil. HCl	1	10	10	10.76
	Na_2O_2 fusion; cake dissolved in dil. HCl	1	2	10	11.10

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

TABLE 5(d)

Summary of analytical methods for Mn in CZN-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	HNO_3 + HF + $HClO_4$; sample diluted with H_2O after fuming	2	9,38	20	0.22
	HNO_3 + HF + $HClO_4$; final soln dilute HCl	2	23,34	30	0.23
	HNO_3 + HF + HCl + $HClO_4$; final soln 1% v/v HCl-10% v/v HNO_3	1	15	10	0.21
	HNO_3 + HF + HCl; final soln 1% v/v HNO_3 containing EDTA	1	40	12	0.23
	HNO_3 + HCl + $HClO_4$; final soln 10% v/v HCl	1	14	10	0.21
	HNO_3 + Br_2 + HCl; final soln 5% v/v HCl	1	16	10	0.23
	HNO_3 + HBr; final soln H_2O	1	22	10	0.20
	HNO_3 + Br_2; no other details	1	35	8	0.20
	HNO_3 + HCl + HF + Br_2; no other details	1	32	10	0.23
	HNO_3 + HCl; residue fused with $K_2S_2O_7$, solns combined	1	19	10	0.22
	Mixed acids; residue fused with Na_2O_2, solns combined	1	10	10	0.21
	HCl + HF + $HClO_4$; final soln dilute HCl + KCl	1	6	10	0.21
	Other details not given	2	5,18	15	0.22
Colorimetric (spectrophotometric)					
Permanganate	Br_2 + HNO_3 + $HClO_4$; insol. fused with Na_2CO_3; periodate as oxidant	2	3,21	20	0.24
Titrimetric					
	HNO_3 + HCl + H_2SO_4; insol. fused with $K_2S_2O_7$; bismuthate as oxidant, reduction with Fe(II), titration with $KMnO_4$	1	29	10	0.20
Emission spectrographic		1	39	5	0.23

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

TABLE 5(e)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	HNO_3 + HCl (some also with HF, Br_2); final soln dilute HNO_3	4	10,26,31,40	42	7.48
	HNO_3 + HCl + $HClO_4$; Pb + Fe ppt'd with NH_3 + $(NH_4)_2CO_3$, dissolved in dilute HNO_3	1	1	10	7.33
	Na_2O_2 fusion; final soln dilute HCl	2	2,15	20	7.37
	HNO_3 + Br_2 + HCl; final soln dilute HCl	1	16	10	7.72
	HCl + HF + $HClO_4$; final soln dilute HCl or $HClO_4$	2	9,23	20	7.42
	HCl + $HClO_4$; final soln dilute $HClO_4$ + HNO_3 + NH_4F	1	34	10	7.42
	Various oxidizing acid mixtures; other details not given	5	6,21,22,32 38	50	7.52
Titrimetric					
EDTA	HNO_3 + HCl + HF + $HClO_4$ + H_2SO_4; Pb ext'd as diethyldithiocarbamate	3	1,3,45	30	7.43
	HNO_3 + H_2SO_4 (one with HCl); Pb sep'd as $PbSO_4$	2	5,35	18	7.51
	HNO_3 + HCl + H_2SO_4; Pb sep'd as $PbSO_4$; titrated potentiometrically	1	16	10	7.50
	Details not given	1	18	12	7.29
Molybdate	HNO_3 + H_2SO_4 (one with HCl); Pb sep'd as $PbSO_4$, dissolved in acetate medium	2	33,34	20	7.42
	HNO_3 + $KClO_3$ + HBr; Pb sep'd as sulphide	1	30	10	7.36
	Details not given	2	13,14	20	7.58
Dichromate	HNO_3 + HCl + H_2SO_4; Pb sep'd as $PbSO_4$; re-ppt'd as chromate, dissolved in HCl and titrated iodometrically	1	24	10	7.45
Gravimetric					
	HNO_3 + HCl + H_2SO_4; Pb sep'd as $PbSO_4$; re-ppt'd as chromate	2	29,39	15	7.19

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

TABLE 5(f)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic Absorption					
	HNO_3; sample soln contained tartaric acid	2	9,21	20	0.059
	$HCl + KClO_3$; sample soln contained tartaric acid	1	6	10	0.055
	$HNO_3 + HCl$; other details not given	3	10,30,35	32	0.053
	$HNO_3 + HF$; sample diluted to 10% HNO_3	1	5	10	0.047
	$HNO_3 + HCl + HClO_4$; sample diluted to 10% HCl	1	14	10	0.057
	$HNO_3 + HF + HClO_4$; residue fused with Na_2CO_3; solns combined	1	34	10	0.048
	$HNO_3 + H_2SO_4$; Sb sep'd by distillation	1	34	10	0.048
	Na_2O_2 fusion; Sb ext'd with MIBK and direct atomization	1	24	10	0.050
Colorimetric (spectrophotometric)					
Rhodamine B	Na_2O_2 fusion; cake dissolved in HCl + tartaric acid, Sb ext'd as xanthate	1	23	10	0.053
Iodide	$HNO_3 + HCl + H_2SO_4$; Sb collected with $Fe(OH)_3$; ext'd as xanthate	1	3	10	0.054
Emission spectrographic		1	40	10	0.046

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

TABLE 5(g)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Titrimetric					
EDTA	HNO_3 + HCl + H_2SO_4 or $HClO_4$ (some also with one or more of Br_2, HBr, HF); Zn sep'd from matrix elements by extn with MIBK (see Appendix B)	12	1,4,6,18,22,24 31,33,34,38,40	133	44.76
	Decomposition as above; R_2O_3 elements removed with NH_3 or NaOH	4	5,16a,39,39	30	44.78
	Decomposition as above; Zn sep'd from matrix elements by anion exchange	1	45	10	44.33
	HNO_3 (fuming); Fe and Mn removed with NH_3, Cd and Cu as sulphides	1	35	12	44.82
Ferrocyanide	HNO_3 + HCl + H_2SO_4 (some also with Br_2 or $KClO_4$); Fe removed with NH_3, Cu removed with Pb° or Al°; molybdate or uranyl acetate as external indicator (see Appendix B)	6	6,9,21,23,30,37	60	44.74
	Decomposition as above; Zn sep'd as sulphide	2	29,33	17	44.81
	No details given	2	13,14	20	44.85
Ferrocyanide-amperometric	Br_2 in CCl_4 + HNO_3 + HCl + H_2SO_4; Fe removed with NH_3, Cu removed with Pb°; Zn treated amperometrically	1	45	10	44.33
Ferrocyanide-potentiometric	HNO_3 + HBr + $HClO_4$; Cu removed with Pb°; Fe titrated with dichromate then Zn titrated potentiometrically	1	16	10	44.78
Atomic absorption					
	Na_2O_2 fusion; cake dissolved in dilute HCl; Na added to std solns; each detn av of 5 readings; air-acetylene	1	2	10	44.79
	HNO_3 + HCl + $HClO_4$; soln made up to 1% HCl, 10% HNO_3; air-acetylene	2	15,19	20	44.89
	HNO_3 + HCl + HF, no other details	2	26,32	20	44.74
Polarographic					
	HNO_3 + HCl + H_2SO_4; polarographed in NH_3-NH_4Cl medium	1	10	10	44.48

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	13	134	0.25	16	3.0
MgO						
	AA	14	150	0.32	10	1.6
SiO_2						
	AA	4	40	1.01	20	2.8
	GRAV	16	146	1.00	21	3.4
				(µg/g)		
Au						
	AA	1	10	0.04	--	12
	FA-AA	3	39	0.10	200	12
	FA	1	10	0.07	--	--
Bi						
	AA	10	108	34	71	7
	COLOR	2	20	27	--	13
	ES	2	15	22	--	19
In						
	AA	6	52	76	39	7
	COLOR	1	10	88	--	5
	ES	2	15	122	--	12
Se						
	AA	4	38	5	74	11
	COLOR	5	50	5	53	7
Sn						
	AA	1	19	93	--	11
	COLOR	2	20	64	--	5
	ES	3	19	66	270	8
	POLAR	1	10	72	--	4
Te						
	AA	1	10	0.2	--	22

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

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	92.8000	2.4900	5	93.2000	3.8341	A	10	93.0000	3.0551	3.29
LAB- 5	(FA)	5	90.6400	1.7430	5	90.5000	1.3096	A	10	90.5700	1.4553	1.61
LAB- 6	(FA)	5	92.2600	1.7855	5	92.7400	2.8858	A	10	92.5000	2.2764	2.46
LAB- 9	(FA-AA)	5	83.6600	1.8623	5	84.3400	1.8623	A	10	84.0000	1.7920	2.13
LAB-10	(AA)	5	93.6400	.4159	5	93.8200	.3421	A	10	93.7300	.3713	.40
LAB-12	(FA-AA)	5	92.4400	1.1950	5	93.1400	.9940	A	10	92.7900	1.0999	1.19
LAB-12	(FA-AA)	5	91.3800	1.3330	5	94.4000	1.5875	REJECT	10	92.8900	2.1079	2.27
LAB-14	(FA)	5	92.0000	.3082	5	93.8200	2.9978	A	10	92.9100	2.2263	2.40
LAB-15	(AA)	5	94.3884	2.9555	5	93.9876	2.1723	A	10	94.1880	2.4544	2.61
LAB-16	(FA-AA)	5	88.5800	.5891	5	92.2600	.3362	REJECT	10	90.4200	1.9915	2.20
LAB-16	(AA)	5	82.2246	2.4313	5	80.8218	1.4549	A	10	81.5232	2.0285	2.49
LAB-18	(FA)	4	90.7250	.8958	4	92.7000	1.4445	A	8	91.7125	1.5338	1.67
LAB-19	(AA)	5	98.0000	1.0000	5	99.2000	1.3038	A	10	98.6000	1.2649	1.28
LAB-21	(FA)	5	95.8000	1.6432	5	95.0000	2.0000	A	10	95.4000	1.7764	1.86
LAB-24	(AA)	5	97.2000	.4472	5	97.0000	0.0000	A	10	97.1000	.3162	.33
LAB-26	(AA)	5	82.4000	.8944	5	81.4000	1.4748	A	10	81.9000	1.2649	1.54
LAB-30	(AA)	5	88.8000	.4472	5	89.8000	.4472	REJECT	10	89.3000	.6749	.76
LAB-31	(AA)	5	100.0000	0.0000	5	92.0000	4.4721	***R**	10	96.0000	5.1640	5.38
LAB-34	(FA)	5	97.1000	2.7810	5	94.8440	1.8228	A	10	95.9720	2.5155	2.62
LAB-35	(AA)	2	95.5000	.7071	2	96.0000	1.4142	A	4	95.7500	.9574	1.00
LAB-37	(FA)	5	97.4400	.3286	5	97.4400	.2191	A	10	97.4400	.2633	.27
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	80.6000	13.6675	16.96
LAB-40	(AA)	5	102.0000	2.7386	5	102.0000	2.7386	A	10	102.0000	2.5820	2.53

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	.2960	.0089	4	.2900	.0115	A	9	.2933	.0100	3.41
LAB- 4	(AA)	5	.2716	.0052	5	.2666	.0059	A	10	.2691	.0058	2.17
LAB- 5	(AA)	5	.2580	.0045	5	.2580	.0045	A	10	.2580	.0042	1.63
LAB- 6	(AA)	5	.2700	.0000	5	.2560	.0055	***R**	10	.2630	.0082	3.13
LAB- 9	(AA)	5	.2520	.0084	5	.2100	.0173	REJECT	10	.2310	.0256	11.07
LAB-10	(AA)	5	.2518	.0015	5	.2516	.0011	A	10	.2517	.0013	.50
LAB-12	(AA)	5	.2328	.0023	5	.2332	.0027	A	10	.2330	.0024	1.01
LAB-14	(AA)	5	.2494	.0015	5	.2482	.0054	A	10	.2488	.0038	1.51
LAB-18	(AA)	6	.2483	.0075	6	.2500	.0063	A	12	.2492	.0067	2.68
LAB-19	(AA)	5	.1700	.0000	5	.1720	.0045	A	10	.1710	.0032	1.85
LAB-21	(AA)	5	.2760	.0055	5	.2720	.0045	A	10	.2740	.0052	1.88
LAB-23	(AA)	10	.2400	.0141	10	.2380	.0132	A	20	.2390	.0133	5.58
LAB-26	(AA)	5	.2700	.0071	5	.2700	.0071	A	10	.2700	.0067	2.47
LAB-34	(AA)	5	.2266	.0038	5	.2290	.0054	A	10	.2278	.0046	2.01
LAB-35	(AA)	2	.1450	.0071	2	.1600	.0141	A	4	.1525	.0126	8.25
LAB-38	(AA)	5	.2100	.0078	5	.2140	.0054	A	10	.2120	.0067	3.15

TABLE 7 (cont'd)

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	.0284	.0000	5	.0283	.0001	A	10	.0284	.0001	.41
LAB- 5	(COLOR)	5	.0200	0.0000	5	.0200	0.0000	***R**	10	.0200	.0000	.00
LAB- 9	(COLOR)	5	.0278	.0027	5	.0312	.0029	A	10	.0295	.0032	10.87
LAB-10	(TITR)	5	.0270	.0010	5	.0274	.0009	A	10	.0272	.0009	3.38
LAB-13	(TITR)	5	.0338	.0013	5	.0338	.0015	A	10	.0338	.0013	3.90
LAB-14		3	.0240	0.0000	3	.0250	.0010	A	6	.0245	.0008	3.41
LAB-15	(COLOR)	5	.0322	.0004	5	.0304	.0038	A	10	.0313	.0027	8.66
LAB-16	(AA)	5	.0259	.0003	5	.0249	.0012	A	10	.0254	.0010	3.83
LAB-18	(COLOR)	3	.0300	.0020	2	.0300	0.0000	A	5	.0300	.0014	4.71
LAB-21	(TITR)	5	.0238	.0004	5	.0238	.0011	A	10	.0238	.0008	3.31
LAB-23	(COLOR)	7	.0227	.0023	7	.0236	.0030	A	14	.0231	.0026	11.35
LAB-24	(COLOR)	5	.0292	.0021	5	.0280	.0014	A	10	.0286	.0018	6.26
LAB-24	(NAA)	5	.0266	.0006	5	.0280	.0027	A	10	.0273	.0020	7.31
LAB-30	(AA)	5	.0184	.0009	5	.0170	.0000	***R**	10	.0177	.0009	5.36
LAB-32	(AA)	5	.0190	.0022	5	.0186	.0035	A	10	.0188	.0028	14.79
LAB-33	(TITR)	5	.0262	.0027	5	.0254	.0017	A	10	.0258	.0021	8.33
LAB-34	(COLOR)	5	.0212	.0014	5	.0215	.0006	A	10	.0214	.0010	4.68
LAB-35	(TITR)	2	.0200	0.0000	2	.0200	0.0000	***R**	4	.0200	0.0000	0.00
LAB-38	(AA)	5	.0301	.0011	5	.0274	.0016	REJECT	10	.0288	.0019	6.69
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.0334	.0023	6.89
LAB-40	(COLOR)	5	.0273	.0012	5	.0273	.0012	A	10	.0273	.0011	4.14

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	.1268	.0004	5	.1278	.0008	REJECT	10	.1273	.0008	.65
LAB- 5	(AA)	5	.1300	.0000	5	.1300	.0000	A	10	.1300	.0000	.00
LAB- 6	(AA)	5	.1288	.0013	5	.1294	.0011	A	10	.1291	.0012	.91
LAB- 9	(AA)	5	.1284	.0021	5	.1288	.0011	A	10	.1286	.0016	1.23
LAB-10	(AA)	5	.1314	.0034	5	.1304	.0021	A	10	.1309	.0027	2.05
LAB-13	(AA)	5	.1260	.0055	5	.1240	.0055	A	10	.1250	.0053	4.22
LAB-14	(AA)	5	.1358	.0016	5	.1364	.0018	A	10	.1361	.0017	1.22
LAB-16	(AA)	5	.1326	.0017	5	.1322	.0018	A	10	.1324	.0016	1.24
LAB-18	(AA)	4	.1365	.0034	4	.1320	0.0000	***R**	8	.1343	.0033	2.45
LAB-19	(AA)	5	.1264	.0004	5	.1269	.0004	A	10	.1267	.0005	.37
LAB-21	(AA)	5	.1388	.0008	5	.1380	.0000	A	10	.1384	.0007	.51
LAB-22	(AA)	5	.1300	.0000	5	.1300	.0000	A	10	.1300	.0000	.00
LAB-23	(AA)	10	.1300	.0000	10	.1300	.0000	A	20	.1300	.0000	.00
LAB-29	(AA)	5	.1200	.0000	5	.1200	.0000	A	10	.1200	.0000	.00
LAB-30	(AA)	5	.1384	.0005	5	.1378	.0004	A	10	.1381	.0006	.41
LAB-32	(AA)	5	.1402	.0008	5	.1388	.0004	REJECT	10	.1395	.0010	.70
LAB-33	(AA)	5	.1398	.0011	5	.1404	.0005	A	10	.1401	.0009	.62
LAB-34	(AA)	5	.1352	.0004	5	.1336	.0005	REJECT	10	.1344	.0010	.72
LAB-35	(AA)	6	.1358	.0053	6	.1344	.0024	A	12	.1351	.0040	2.94
LAB-38	(AA)	5	.1280	.0026	5	.1288	.0029	A	10	.1284	.0027	2.08
LAB-38	(XRF)	5	.1308	.0008	5	.1306	.0009	A	10	.1307	.0008	.61
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.1302	.0072	5.55
LAB-40	(AA)	6	.1350	.0055	6	.1354	.0051	A	12	.1352	.0051	3.74

TABLE 7 (cont'd)

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	.1534	.0024	5	.1514	.0005	A	10	.1524	.0020	1.28
LAB- 1	(AA)	5	.1434	.0025	5	.1430	.0025	A	10	.1432	.0024	1.67
LAB- 1	(COLOR)	5	.1446	.0018	5	.1454	.0021	A	10	.1450	.0019	1.30
LAB- 2	(AA)	5	.1364	.0009	5	.1354	.0011	A	10	.1359	.0011	.81
LAB- 3	(COLOR)	5	.1518	.0008	5	.1532	.0015	A	10	.1525	.0014	.89
LAB- 5	(AA)	5	.1300	.0000	5	.1300	.0000	A	10	.1300	.0000	.00
LAB- 6	(AA)	5	.1360	.0022	5	.1360	.0022	A	10	.1360	.0021	1.55
LAB- 9	(AA)	5	.1436	.0015	5	.1428	.0008	A	10	.1432	.0012	.86
LAB-10	(AA)	5	.1386	.0023	5	.1392	.0022	A	10	.1389	.0021	1.53
LAB-12	(AA)	5	.1438	.0004	5	.1434	.0005	A	10	.1436	.0005	.36
LAB-14	(AA)	5	.1392	.0008	5	.1398	.0013	A	10	.1395	.0011	.77
LAB-18	(AA)	6	.1550	.0055	3	.1600	0.0000	A	9	.1567	.0050	3.19
LAB-19	(AA)	5	.1434	.0013	5	.1448	.0008	A	10	.1441	.0013	.89
LAB-21	(AA)	5	.1432	.0008	5	.1422	.0004	REJECT	10	.1427	.0008	.58
LAB-22	(AA)	5	.1500	.0000	5	.1500	.0000	A	10	.1500	.0000	.00
LAB-23	(AA)	10	.1490	.0032	10	.1480	.0042	A	20	.1485	.0037	2.47
LAB-24	(AA)	5	.1380	.0045	5	.1340	.0055	A	10	.1360	.0052	3.80
LAB-26	(AA)	5	.1476	.0023	5	.1484	.0022	A	10	.1480	.0022	1.46
LAB-29	(AA)	5	.1380	.0045	5	.1380	.0045	A	10	.1380	.0042	3.06
LAB-30	(AA)	5	.1304	.0054	5	.1360	.0041	A	10	.1332	.0054	4.05
LAB-31	(AA)	5	.1600	0.0000	5	.1580	.0045	A	10	.1590	.0032	1.99
LAB-32	(AA)	5	.1422	.0011	5	.1396	.0011	REJECT	10	.1409	.0017	1.23
LAB-34	(AA)	5	.1450	.0010	5	.1462	.0004	REJECT	10	.1456	.0010	.66
LAB-35	(AA)	6	.1433	.0052	6	.1450	.0055	A	12	.1442	.0051	3.57
LAB-38	(AA)	4	.1525	.0019	5	.1518	.0016	A	9	.1521	.0017	1.11
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	.1380	.0110	7.94
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	.0700	.0000	.00
LAB-40	(AA)	6	.1451	.0027	6	.1449	.0027	A	12	.1450	.0025	1.76

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	10.7980	.0390	5	10.7600	.0339	A	10	10.7790	.0398	.37
LAB- 1	(AA)	5	10.9560	.0680	5	11.0420	.0421	REJECT	10	10.9990	.0700	.64
LAB- 2	(AA)	5	11.0480	.0268	5	11.1560	.1004	REJECT	10	11.1020	.0897	.81
LAB- 5	(TITR)	5	11.0860	.0134	5	11.1040	.0134	A	10	11.0950	.0158	.14
LAB- 6	(TITR)	5	11.0420	.0217	5	11.0320	.0383	A	10	11.0370	.0298	.27
LAB- 9	(TITR)	5	10.7500	.0224	5	10.7700	.0548	A	10	10.7600	.0408	.38
LAB-10	(AA)	5	10.7560	.0434	5	10.7720	.0610	A	10	10.7640	.0506	.47
LAB-12	(AA)	5	10.9920	.0335	5	11.0000	.0400	A	10	10.9960	.0350	.32
LAB-13	(TITR)	5	10.8840	.0110	5	10.8800	.0110	A	10	10.8820	.0105	.10
LAB-14	(TITR)	5	11.0100	.0224	5	11.0000	.0274	A	10	11.0050	.0242	.22
LAB-15	(AA)	5	10.9820	.0548	5	11.0020	.0837	A	10	10.9920	.0675	.61
LAB-16	(TITR)	5	10.9000	.0402	5	10.8400	.0482	A	10	10.8700	.0525	.48
LAB-18	(TITR)	6	11.1650	.0315	6	11.1917	.0714	A	12	11.1783	.0544	.49
LAB-19	(AA)	5	12.0220	.0228	5	12.0460	.0152	A	10	12.0340	.0222	.18
LAB-21	(TITR)	5	10.9120	.0084	5	10.9040	.0219	A	10	10.9080	.0162	.15
LAB-22	(AA)	5	10.9000	.0000	5	10.9600	.0548	***R**	10	10.9300	.0483	.44
LAB-23	(TITR)	5	10.9600	.0000	5	10.9400	.0274	A	10	10.9500	.0211	.19
LAB-24	(TITR)	5	10.8200	.0100	5	10.8360	.0134	A	10	10.8280	.0140	.13
LAB-26	(TITR)	5	10.7600	.0894	5	10.7800	.0447	A	10	10.7700	.0675	.63
LAB-29	(TITR)	5	11.1600	.0548	5	11.1400	.0548	A	10	11.1500	.0527	.47
LAB-30	(TITR)	5	10.6700	.0612	5	10.7460	.1258	A	10	10.7080	.1015	.95
LAB-31	(TITR)	5	10.9040	.0251	5	10.9100	.0406	A	10	10.9070	.0320	.29
LAB-32	(AA)	5	11.4480	.0438	5	11.3720	.0383	REJECT	10	11.4100	.0558	.49
LAB-34	(TITR)	5	10.8320	.0110	5	10.8600	.0245	REJECT	10	10.8460	.0232	.21
LAB-35	(TITR)	2	10.9050	.0212	2	10.9200	.0424	A	4	10.9125	.0287	.26
LAB-38	(AA)	5	11.1780	.0879	5	11.2260	.0518	A	10	11.2020	.0725	.65
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	10.9240	.0358	.33
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	11.0040	.0089	.08
LAB-40	(AA)	6	10.7083	.0801	6	10.7750	.0689	A	12	10.7417	.0793	.74

TABLE 7 (cont'd)

Mg (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	45.6600	.9711	5	44.9800	.8468	A	10	45.3200	.9307	2.05
LAB- 5	(AA)	5	48.8000	.5657	5	46.9600	.6066	REJECT	10	47.8800	1.1163	2.33
LAB- 6	(AA)	5	42.8000	1.9235	5	43.6000	.5477	A	10	43.2000	1.3984	3.24
LAB- 9	(AA)	5	38.6000	.8944	5	40.6000	1.3416	REJECT	10	39.6000	1.5055	3.80
LAB-10	(AA)	5	45.8000	2.3875	5	45.4000	2.0736	A	10	45.6000	2.1187	4.65
LAB-14	(AA)	5	35.5000	1.5411	5	32.8000	3.1937	A	10	34.1500	2.7593	8.08
LAB-15	(AA)	5	28.4568	7.2809	5	36.0720	2.4544	A	10	32.2644	6.5074	20.17
LAB-18	(AA)	4	58.4500	.7141	3	57.5333	.8505	A	7	58.0571	.8580	1.48
LAB-22	(AA)	5	49.0000	2.1213	5	47.2000	2.0494	A	10	48.1000	2.1833	4.54
LAB-23	(AA)	THERE IS ONLY 1 BOTTLE							10	57.1000	2.6013	4.56
LAB-24	(AA)	5	42.0000	3.3912	5	39.8000	.8367	A	10	40.9000	2.6013	6.36
LAB-30	(AA)	5	42.3000	.6595	5	42.3000	.2739	A	10	42.3000	.4761	1.13
LAB-34	(COLOR)	5	40.7600	.7537	5	39.3400	.7829	REJECT	10	40.0500	1.0416	2.60
LAB-35	(AA)	2	45.5000	.7071	2	46.0000	2.8284	A	4	45.7500	1.7078	3.73
LAB-38	(AA)	5	45.2000	1.0700	5	39.1200	1.5691	REJECT	10	42.1600	3.4455	8.17
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	45.0000	11.1803	24.85
LAB-40	(AA)	5	41.1100	1.0449	5	40.6600	.3831	A	10	40.8850	.7789	1.91

Mn (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	.2380	.0023	5	.2364	.0009	A	10	.2372	.0019	.79
LAB- 5	(AA)	5	.2180	.0045	5	.2200	.0000	A	10	.2190	.0032	1.44
LAB- 6	(AA)	5	.2116	.0009	5	.2124	.0009	A	10	.2120	.0009	.44
LAB- 9	(AA)	5	.2182	.0025	5	.2154	.0011	A	10	.2168	.0023	1.08
LAB-10	(AA)	5	.2068	.0025	5	.2074	.0029	A	10	.2071	.0026	1.24
LAB-14	(AA)	5	.2056	.0022	5	.2064	.0030	A	10	.2060	.0025	1.23
LAB-15	(AA)	5	.2108	.0066	5	.2120	.0083	A	10	.2114	.0071	3.37
LAB-16	(AA)	5	.2344	.0036	5	.2290	.0021	REJECT	10	.2317	.0040	1.71
LAB-18	(AA)	3	.2223	.0042	2	.2275	.0035	A	5	.2244	.0044	1.98
LAB-19	(AA)	5	.2154	.0009	5	.2160	.0010	A	10	.2157	.0009	.44
LAB-21	(COLOR)	5	.2400	.0000	5	.2420	.0045	A	10	.2410	.0032	1.31
LAB-22	(AA)	5	.2000	.0000	5	.2000	.0000	A	10	.2000	.0000	.00
LAB-23	(AA)	10	.2310	.0032	10	.2310	.0032	A	20	.2310	.0031	1.33
LAB-29	(TITR)	5	.1980	.0045	5	.1980	.0045	A	10	.1980	.0042	2.13
LAB-32	(AA)	5	.2300	.0007	5	.2294	.0009	A	10	.2297	.0008	.36
LAB-34	(AA)	5	.2234	.0011	5	.2238	.0019	A	10	.2236	.0015	.67
LAB-35	(AA)	4	.1946	.0127	4	.1985	.0107	A	8	.1965	.0110	5.61
LAB-38	(AA)	5	.2188	.0029	5	.2190	.0014	A	10	.2189	.0022	1.00
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.2268	.0107	4.70
LAB-40	(AA)	6	.2278	.0076	6	.2283	.0071	A	12	.2280	.0070	3.08

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	7.4480	.0205	5	7.4220	.0356	A	10	7.4350	.0306	.41
LAB- 1	(AA)	5	7.3240	.0658	5	7.3340	.0602	A	10	7.3290	.0597	.81
LAB- 2	(AA)	5	7.2840	.0555	5	7.2920	.0438	A	10	7.2880	.0473	.65
LAB- 3	(TITR)	5	7.4278	.0097	5	7.3708	.0141	REJECT	10	7.3993	.0321	.43
LAB- 5	(TITR)	5	7.6060	.0230	5	7.5760	.0329	A	10	7.5910	.0311	.41
LAB- 6	(AA)	5	7.4680	.0444	5	7.4500	.0453	A	10	7.4590	.0433	.58
LAB- 9	(AA)	5	7.3000	.1000	5	7.4200	.2490	A	10	7.3600	.1897	2.58
LAB-10	(AA)	5	7.4600	.0071	5	7.4620	.0164	A	10	7.4610	.0120	.16
LAB-13	(TITR)	5	7.6810	.0089	5	7.6730	.0110	A	10	7.6770	.0103	.13
LAB-14	(TITR)	5	7.4820	.0455	5	7.4700	.0274	A	10	7.4760	.0360	.48
LAB-15	(AA)	5	7.4330	.0432	5	7.4690	.0472	A	10	7.4510	.0467	.63
LAB-16	(TITR)	5	7.5060	.0488	5	7.4860	.0167	A	10	7.4960	.0360	.48
LAB-16	(AA)	5	7.7070	.0110	5	7.7374	.0554	A	10	7.7222	.0409	.53
LAB-18	(TITR)	6	7.2917	.0671	6	7.2950	.0689	A	12	7.2933	.0649	.89
LAB-19	(AA)	5	6.8220	.0311	5	6.7860	.0219	A	10	6.8040	.0317	.47
LAB-21	(AA)	5	7.5900	.0000	5	7.4200	.0255	***R**	10	7.5050	.0912	1.22
LAB-22	(AA)	5	7.4860	.0378	5	7.4960	.0055	A	10	7.4910	.0260	.35
LAB-23	(AA)	5	7.4800	.0447	5	7.4600	.0548	A	10	7.4700	.0483	.65
LAB-24	(TITR)	5	7.4520	.0164	5	7.4460	.0251	A	10	7.4490	.0202	.27
LAB-26	(AA)	5	7.4700	.1255	5	7.5460	.1307	A	10	7.5080	.1273	1.70
LAB-29	(GRAV)	5	7.2620	.0383	5	7.2860	.0378	A	10	7.2740	.0381	.52
LAB-30	(TITR)	5	7.3500	.0274	5	7.3600	.0418	A	10	7.3550	.0337	.46
LAB-31	(AA)	5	7.3600	.0406	5	7.3300	.0173	A	10	7.3450	.0334	.45
LAB-32	(AA)	5	7.3980	.0545	5	7.4360	.0472	A	10	7.4170	.0521	.70
LAB-33	(TITR)	5	7.3840	.0422	5	7.4100	.0863	A	10	7.3970	.0655	.89
LAB-34	(TITR)	5	7.4280	.0205	5	7.4480	.0356	A	10	7.4380	.0294	.39
LAB-34	(AA)	5	7.4280	.0455	5	7.4080	.0303	A	10	7.4180	.0379	.51
LAB-35	(TITR)	4	7.4100	.0424	4	7.4200	.0392	A	8	7.4150	.0382	.51
LAB-38	(AA)	5	7.7240	.0483	5	7.6840	.0261	A	10	7.7040	.0422	.55
LAB-38	(XRF)	5	7.9440	.0288	5	7.8900	.0524	A	10	7.9170	.0490	.62
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	7.0340	.2063	2.93
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	6.8340	.2428	3.55
LAB-40	(AA)	6	7.5633	.2439	6	7.5917	.2417	A	12	7.5775	.2320	3.06
LAB-45	(TITR)	5	7.4500	.0100	5	7.4520	.0148	A	10	7.4510	.0120	.16

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	30.3980	.0540	5	30.3960	.0546	A	10	30.3970	.0512	.17
LAB- 6	(GRAV)	5	29.7400	.1079	5	29.6240	.3293	A	10	29.6820	.2390	.81
LAB- 9	(GRAV)	5	29.5800	.0430	5	29.6160	.0760	A	10	29.5980	.0612	.21
LAB-10	(GRAV)	5	30.2080	.0701	5	30.2060	.0564	A	10	30.2070	.0600	.20
LAB-13	(GRAV)	5	30.3190	.0295	5	30.3950	.0365	REJECT	10	30.3570	.0508	.17
LAB-14	(COMB)	5	31.5000	.2318	5	31.5600	.2770	A	10	31.5300	.2429	.77
LAB-14	(GRAV)	5	30.3020	.1169	5	30.3060	.1466	A	10	30.3040	.1250	.41
LAB-15	(GRAV)	5	30.6210	.2888	5	30.8618	.2650	A	10	30.7414	.2905	.94
LAB-18	(GRAV)	6	30.2400	.0998	6	30.1850	.0683	A	12	30.2125	.0865	.29
LAB-21	(GRAV)	5	30.0860	.0730	5	30.2640	.0598	REJECT	10	30.1750	.1130	.37
LAB-23	(GRAV)	5	29.9280	.1028	4	29.8600	.0594	A	9	29.8978	.0889	.30
LAB-26	(COMB)	5	31.8200	.5404	5	31.8400	.4561	A	10	31.8300	.4715	1.48
LAB-30	(GRAV)	5	27.9000	.2739	5	28.7000	.5523	REJECT	10	28.3000	.5888	2.08
LAB-31	(GRAV)	5	29.6680	.0606	5	29.5840	.0709	A	10	29.6260	.0763	.26
LAB-34	(GRAV)	5	30.7140	.0814	5	30.7260	.3316	A	10	30.7200	.2277	.74
LAB-35	(GRAV)	2	30.6900	0.0000	2	30.4600	.0849	A	4	30.5750	.1415	.46
LAB-38	(GRAV)	5	30.1220	.0545	5	30.2000	.1125	A	10	30.1610	.0929	.31
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							4	31.0125	.1284	.41
LAB-39	(GRAV)	THERE IS ONLY 1 BOTTLE							5	31.9320	.5319	1.67

TABLE 7 (cont'd)

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	.0541	.0007	5	.0541	.0007	A	10	.0541	.0007	1.22
LAB- 5	(AA)	5	.0474	.0009	5	.0474	.0005	A	10	.0474	.0007	1.48
LAB- 6	(AA)	5	.0546	.0005	5	.0546	.0015	A	10	.0546	.0011	1.97
LAB- 9	(AA)	5	.0556	.0026	5	.0612	.0033	REJECT	10	.0584	.0041	7.00
LAB-10	(AA)	5	.0518	.0013	5	.0516	.0005	A	10	.0517	.0009	1.83
LAB-14	(AA)	5	.0578	.0011	5	.0570	.0010	A	10	.0574	.0011	1.87
LAB-18	(AA)	2	.0200	0.0000	2	.0200	0.0000	***R***	4	.0200	0.0000	0.00
LAB-19	(AA)	5	.0374	.0015	5	.0361	.0007	A	10	.0368	.0013	3.47
LAB-21	(AA)	5	.0590	.0007	5	.0592	.0004	A	10	.0591	.0006	.96
LAB-23	(COLOR)	5	.0532	.0020	5	.0536	.0013	A	10	.0534	.0016	3.08
LAB-24	(AA)	5	.0489	.0032	5	.0511	.0022	A	10	.0500	.0028	5.62
LAB-30	(AA)	5	.0536	.0017	5	.0534	.0015	A	10	.0535	.0015	2.82
LAB-34	(AA)	5	.0484	.0008	5	.0483	.0015	A	10	.0483	.0011	2.31
LAB-34	(AA)	5	.0468	.0022	5	.0482	.0026	A	10	.0475	.0024	4.99
LAB-35	(AA)	6	.0530	.0032	6	.0518	.0033	A	12	.0524	.0031	6.00
LAB-39	(ES)	THERE IS ONLY 1 BOTTLE							5	.0322	.0044	13.78
LAB-40	(ES)	5	.0458	.0044	5	.0470	.0037	A	10	.0464	.0039	8.39

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	(TITR)	THERE ARE MORE THAN 2 BOTTLES							25	44.7724	.0837	.19
LAB- 2	(AA)	5	44.8040	.1252	5	44.7800	.1175	A	10	44.7920	.1152	.26
LAB- 4	(TITR)	5	44.8240	.0230	5	44.8440	.1011	A	10	44.8340	.0700	.16
LAB- 5	(TITR)	5	44.5520	.0277	5	44.5740	.0365	A	10	44.5630	.0327	.07
LAB- 6	(TITR)	5	44.4600	.0886	5	44.5340	.0899	A	10	44.4970	.0927	.21
LAB- 6	(TITR)	5	44.6320	.1092	5	44.6600	.0485	A	10	44.6460	.0810	.18
LAB- 9	(TITR)	5	44.3360	.0397	5	44.3300	.0200	A	10	44.3330	.0298	.07
LAB-10	(POLAR)	5	44.4880	.0502	5	44.4760	.0607	A	10	44.4820	.0529	.12
LAB-13	(TITR)	5	45.2404	.0553	5	45.1982	.0746	A	10	45.2193	.0658	.15
LAB-14	(TITR)	5	44.4600	.0548	5	44.5000	.0707	A	10	44.4800	.0632	.14
LAB-15	(AA)	5	44.4890	.2243	5	44.5894	.3469	A	10	44.5392	.2804	.63
LAB-16	(TITR)	5	44.7920	.1026	5	44.7700	.1726	A	10	44.7810	.1344	.30
LAB-16	(TITR)	5	45.0180	.0835	5	44.8980	.0687	REJECT	10	44.9580	.0959	.21
LAB-16	(TITR)	4	45.5310	.0744	4	45.4635	.0263	A	8	45.4973	.0630	.14
LAB-18	(TITR)	6	45.2050	.0446	6	45.2183	.0755	A	12	45.2117	.0595	.13
LAB-19	(AA)	5	45.1760	.3723	5	45.3000	.2092	A	10	45.2380	.2921	.65
LAB-21	(TITR)	5	44.6340	.0488	5	44.6340	.0378	A	10	44.6340	.0412	.09
LAB-22	(TITR)	5	44.6000	.0707	5	44.5600	.0548	A	10	44.5800	.0632	.14
LAB-23	(TITR)	5	45.0200	.0837	5	45.0400	.0894	A	10	45.0300	.0823	.18
LAB-24	(TITR)	5	44.9840	.0976	5	44.9800	.0959	A	10	44.9820	.0913	.20
LAB-24	(TITR)	5	44.5500	.0975	5	44.5840	.1218	A	10	44.5670	.1055	.24
LAB-26	(AA)	5	44.2800	.2280	5	44.2000	.2449	A	10	44.2400	.2271	.51
LAB-29	(TITR)	5	44.7800	.0837	5	44.8000	.0000	A	10	44.7900	.0568	.13
LAB-30	(TITR)	5	44.8240	.1309	5	44.7640	.0654	A	10	44.7940	.1025	.23
LAB-31	(TITR)	5	45.1820	.1472	4	45.4075	.1352	A	9	45.2822	.1784	.39
LAB-32	(AA)	5	45.3220	.0998	5	45.1720	.1657	A	10	45.2470	.1513	.33
LAB-33	(TITR)	3	44.8133	.1155	4	44.8650	.0705	A	7	44.8429	.0877	.20
LAB-33	(TITR)	INSUFFICIENT DATA							3	44.7800	.0872	.19
LAB-34	(TITR)	5	44.5460	.0439	5	44.6360	.0313	REJECT	10	44.5910	.0595	.13
LAB-35	(TITR)	6	44.8267	.0971	6	44.8217	.1114	A	12	44.8242	.0997	.22
LAB-37	(TITR)	5	44.9800	.0765	5	44.9700	.1283	A	10	44.9750	.0997	.22
LAB-38	(TITR)	5	44.5580	.0981	5	44.3580	.0709	REJECT	10	44.4580	.1327	.30
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	44.3980	.2062	.46
LAB-39	(TITR)	THERE IS ONLY 1 BOTTLE							5	45.2200	.3834	.85
LAB-40	(TITR)	7	44.5857	.2035	7	44.6429	.1397	A	14	44.6143	.1703	.38
LAB-45	(TITR)	5	44.3260	.0598	5	44.3420	.0164	A	10	44.3340	.0422	.10
LAB-45	(TITR)	5	44.3240	.0358	5	44.3440	.0477	A	10	44.3340	.0412	.09

TABLE 8

Analytical results for reference concentrate CZN-1 †

Ag (µg/g)

LAB-10 (AA)	94	94	94	93	94	94	93	94	94	94
LAB-15 (AA)	92	93	92	95	99	95	96	91	92	95
LAB-16 (AA)	81	80	80	86	84	80	80	80	80	83
LAB-19 (AA)	98	97	99	97	99	97	100	99	100	100
LAB-24 (AA)	98	97	97	97	97	97	97	97	97	97
LAB-26 (AA)	83	83	82	83	81	83	82	83	81	79
LAB-30 (AA)	88	89	89	89	89	89	90	90	90	90
LAB-31 (AA)	100	100	100	100	100	90	90	100	90	90
LAB-35 (AA)	95	96	97	95						
LAB-40 (AA)	105	100	100	100	105	105	100	100	100	105
*LAB-39 (ES)	70	65	90	80	98					
LAB- 5 (FA)	91	91	93	89	89	91	90	91	89	93
LAB- 6 (FA)	95	94	91	91	91	95	96	91	92	89
LAB-14 (FA)	92	92	92	92	92	93	99	93	92	92
LAB-18 (FA)	90	90	92	92	94	94	92	91		
LAB-21 (FA)	96	93	96	97	97	96	93	94	94	98
LAB-34 (FA)	99	97	95	94	100	94	97	94	93	97
LAB-37 (FA)	97	97	98	97	97	97	97	98	97	97
LAB- 1 (FA-AA)	96	93	89	93	93	89	96	96	89	96
LAB- 9 (FA-AA)	82	82	86	82	86	82	86	86	86	82
LAB-12 (FA-AA)	94	91	93	92	92	93	94	93	92	94
LAB-12 (FA-AA)	92	93	91	91	90	97	93	95	95	93
LAB-16 (FA-AA)	88	88	88	90	89	93	93	93	92	92

Al_2O_3 (wt %)

LAB- 1 (AA)	.30	.30	.30	.30	.28	.30	.36*	.28	.28	.30
LAB- 4 (AA)	.27	.27	.28	.28	.27	.26	.28	.27	.26	.27
LAB- 5 (AA)	.26	.26	.26	.26	.25	.25	.26	.26	.26	.26
LAB- 6 (AA)	.27	.27	.27	.27	.27	.25	.25	.26	.26	.26
LAB- 9 (AA)	.25	.26	.25	.24	.26	.24	.21	.20	.20	.20
LAB-10 (AA)	.25	.25	.25	.25	.25	.25	.25	.25	.25	.25
LAB-12 (AA)	.23	.23	.23	.24	.23	.23	.23	.24	.24	.23
LAB-14 (AA)	.25	.25	.25	.25	.25	.25	.26	.25	.25	.24
*LAB-18 (AA)	.24	.25	.25	.26	.25	.24	.25	.24	.26	.25
	.25	.25								
*LAB-19 (AA)	.17	.17	.17	.17	.17	.17	.18	.17	.17	.17
LAB-21 (AA)	.27	.28	.27	.28	.28	.28	.27	.27	.27	.27
LAB-23 (AA)	.24	.22	.21	.24	.24	.25	.25	.25	.25	.25
	.24	.22	.22	.24	.22	.25	.25	.25	.25	.24
LAB-26 (AA)	.27	.26	.27	.28	.27	.27	.26	.28	.27	.27
LAB-34 (AA)	.22	.22	.23	.23	.23	.23	.22	.23	.23	.24
*LAB-35 (AA)	.14	.15	.15	.17						
LAB-38 (AA)	.21	.20	.22	.21	.22	.22	.21	.21	.22	.21

† See legend and note at end of table on p 30.

TABLE 8 (cont'd)

As (wt %)

LAB-16 (AA)	.026	.026	.026	.026	.026	.024	.027	.025	.025	.025
LAB-30 (AA)	.019	.018	.017	.019	.019	.017	.017	.017	.017	.017
LAB-32 (AA)	.020	.015	.020	.020	.020	.015	.020	.023	.020	.015
LAB-38 (AA)	.031	.031	.030	.029	.029	.029	.027	.026	.026	.029
LAB-10 (TITR)	.028	.026	.026	.028	.027	.027	.027	.027	.029	.027
LAB-13 (TITR)	.036	.033	.033	.033	.034	.036	.034	.032	.033	.034
LAB-21 (TITR)	.024	.024	.024	.023	.024	.024	.024	.025	.024	.022
LAB-33 (TITR)	.029	.029	.025	.023	.025	.025	.025	.023	.027	.027
LAB-35 (TITR)	.020	.020	.020	.020						
LAB- 3 (COLOR)	.028	.029	.028	.028	.028	.028	.028	.028	.029	.028
LAB- 5 (COLOR)	.020	.020	.020	.020	.020	.020	.020	.020	.020	.020
LAB- 9 (COLOR)	.030	.024	.029	.030	.026	.028	.030	.031	.036	.031
LAB-15 (COLOR)	.033	.032	.032	.032	.032	.029	.028	.028	.030	.037
LAB-18 (COLOR)	.028	.032	.030	.030	.030					
LAB-23 (COLOR)	.023	.025	.023	.025	.018	.022	.023	.025	.026	.026
	.026	.020	.019	.023						
LA8-24 (COLOR)	.033	.027	.029	.028	.029	.029	.028	.029	.026	.029
LAB-34 (COLOR)	.019	.021	.020	.023	.023	.022	.022	.022	.021	.021
LAB-40 (COLOR)	.027	.028	.029	.027	.026	.029	.027	.027	.028	.026
LAB-39 (ES)	.033	.031	.032	.037	.034					
LAB-24 (NAA)	.026	.027	.027	.026	.027	.027	.033	.026	.027	.027
LAB-14	.024	.024	.024	.024	.025	.026				

Au (µg/g)

LAB-14 (AA)	.05	.03	.03	.04	.03	.04	.03	.03	.04	.05
LAB- 5 (FA)	.07	.07	.07	.07	.07	.07	.07	.07	.07	.07
LAB- 1 (FA-AA)	.14	.14	.14	.10	.14	.10	.10	.10	.10	.14
	.10	.10	.14	.10	.10	.10	.10	.10	.10	.10
	.10	.10	.14	.10	.10					
LAB- 6 (FA-AA)	.07	.07	.06	.06	.07	.07	.07	.05	.07	.07
LAB-18 (FA-AA)	.14	.14	.16	.14						

Bi (µg/g)

*LAB- 1 (AA)	89	106	95	83	83	89	100	94	105	77
LAB- 6 (AA)	25	25	25	27	25	25	27	25	25	25
*LAB- 9 (AA)	100	110	110	120	140	130	140	120	120	130
LAB-10 (AA)	56	58	56	60	56	61	56	60	58	56
*LAB-13 (AA)	601	601	601	601	601	601	601	601	601	601
LAB-14 (AA)	23	17	15	23	18	20				
LAB-19 (AA)	24	27	27	27	25	27	27	27	26	25
LAB-21 (AA)	70	70	70	60	70	80	70	70	70	70
LAB-23 (AA)	32	29	32	25	32	30	29	27	37	30
	30	32	30	25	30	27	29	33	27	29
LAB-30 (AA)	25	25	25	25	25	25	25	25	25	25
LAB-34 (AA)	24	23	24	24	24	24	23	24	23	24
LAB-35 (AA)	36	30	28	44	36	34	36	35	22	44
	40	26								
LAB-38 (AA)	23	25	23	22	24	24	24	22	22	22
LAB- 3 (COLOR)	23	22	21	21	23	24	24	23	23	23
LAB-24 (COLOR)	30	36	37	49	27	25	31	25	30	32
LAB-39 (ES)	30	35	28	25	20					
LAB-40 (ES)	20	15	20	20	25	20	20	20	15	15

TABLE 8 (cont'd)

CaO (wt %)

*LAB- 1 (AA)	.32	.35	.34	.36	.35	.36	.43*	.34	.36	.36
LAB- 2 (AA)	.31	.31	.31	.31	.31	.30	.30	.30	.30	.30
LAB- 4 (AA)	.28	.26	.24	.24	.24	.25	.24	.25	.24	.25
LAB- 5 (AA)	.22	.23	.23	.22	.22	.22	.22	.22	.22	.22
LAB- 6 (AA)	.24	.24	.24	.24	.24	.24	.24	.24	.24	.24
LAB- 9 (AA)	.21	.21	.21	.22	.21	.22	.27	.27	.25	.26
*LAB-10 (AA)	.26	.26	.25	.26	.26	.26	.25	.25	.25	.26
LAB-12 (AA)	.24	.25	.24	.25	.24	.25	.24	.25	.25	.24
*LAB-13 (AA)	.14	.14	.14	.14	.14	.13	.14	.14	.14	.13
LAB-14 (AA)	.23	.23	.23	.23	.23	.23	.23	.23	.23	.23
*LAB-18 (AA)	.29	.31	.31	.30						
*LAB-19 (AA)	.21	.21	.21	.21	.21	.21	.21	.21	.21	.21
LAB-21 (AA)	.34	.33	.30	.34	.30	.33	.33	.34	.35	.33
LAB-23 (AA)	.23	.24	.24	.23	.23	.24	.25	.25	.25	.25
	.24	.24	.24	.23	.24	.25	.25	.26	.25	.25
LAB-26 (AA)	.22	.23	.21	.22	.22	.21	.23	.23	.23	.23
*LAB-30 (AA)	.20	.20	.21	.20	.21	.21	.21	.21	.21	.20
LAB-34 (AA)	.28	.27	.27	.27	.27	.27	.27	.27	.27	.27
LAB-35 (AA)	.22	.23	.23	.23						
LAB-38 (AA)	.25	.23	.23	.23	.23	.24	.23	.23	.23	.23

Cd (wt %)

LAB- 1 (AA)	.127	.127	.127	.126	.127	.129	.127	.128	.127	.128
LAB- 5 (AA)	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
LAB- 6 (AA)	.127	.130	.127	.130	.130	.130	.130	.130	.128	.130
LAB- 9 (AA)	.131	.128	.130	.126	.127	.127	.129	.129	.129	.130
LAB-10 (AA)	.135	.135	.128	.129	.130	.130	.132	.127	.131	.132
LAB-13 (AA)	.120	.130	.130	.120	.130	.130	.130	.120	.120	.120
LAB-14 (AA)	.138	.134	.135	.137	.135	.138	.135	.138	.137	.134
LAB-16 (AA)	.130	.133	.134	.134	.132	.132	.131	.130	.134	.134
LAB-18 (AA)	.136	.132	.138	.140	.132	.132	.132	.132		
LAB-19 (AA)	.127	.126	.126	.127	.127	.128	.127	.127	.127	.127
LAB-21 (AA)	.139	.139	.140	.138	.138	.138	.138	.138	.138	.138
LAB-22 (AA)	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
LAB-23 (AA)	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
*LAB-29 (AA)	.120	.120	.120	.120	.120	.120	.120	.120	.120	.120
LAB-30 (AA)	.138	.139	.138	.138	.139	.138	.137	.138	.138	.138
LAB-32 (AA)	.141	.141	.139	.140	.140	.138	.139	.139	.139	.139
LAB-33 (AA)	.140	.140	.141	.138	.140	.141	.140	.140	.140	.141
LAB-34 (AA)	.136	.135	.135	.135	.135	.134	.133	.133	.134	.134
LAB-35 (AA)	.140	.127	.136	.143	.134	.136	.132	.131	.136	.134
	.137	.137								
LAB-38 (AA)	.132	.129	.127	.125	.127	.126	.130	.133	.129	.126
LAB-40 (AA)	.130	.130	.140	.140	.140	.130	.133	.130	.140	.140
	.140	.130								
LAB-39 (ES)	.128	.122	.135	.140	.126					
LAB-38 (XRF)	.131	.130	.131	.132	.131	.129	.131	.131	.130	.131

TABLE 8 (cont'd)

Cu (wt %)

LAB- 1 (AA)	.142	.142	.141	.147	.145	.141	.140	.146	.143	.145
LAB- 2 (AA)	.138	.136	.136	.136	.136	.135	.134	.135	.137	.136
LAB- 5 (AA)	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
LAB- 6 (AA)	.135	.135	.135	.140	.135	.135	.135	.135	.135	.140
LAB- 9 (AA)	.146	.143	.143	.144	.142	.143	.142	.144	.142	.143
LAB-10 (AA)	.137	.142	.137	.137	.140	.140	.137	.140	.142	.137
LAB-12 (AA)	.144	.144	.143	.144	.144	.143	.143	.144	.144	.143
LAB-14 (AA)	.140	.139	.138	.140	.139	.140	.141	.141	.138	.139
LAB-18 (AA)	.160	.150	.160	.160	.150	.150	.160	.160	.160	
LAB-19 (AA)	.142	.142	.145	.144	.144	.145	.146	.145	.144	.144
LAB-21 (AA)	.142	.144	.143	.144	.143	.142	.143	.142	.142	.142
LAB-22 (AA)	.150	.150	.150	.150	.150	.150	.150	.150	.150	.150
LAB-23 (AA)	.150	.150	.150	.150	.140	.150	.150	.150	.150	.150
	.150	.150	.150	.140	.140	.150	.150	.150	.150	.150
LAB-24 (AA)	.140	.140	.130	.140	.140	.130	.130	.140	.130	.140
LAB-26 (AA)	.146	.151	.145	.148	.148	.148	.152	.146	.148	.148
LAB-29 (AA)	.130	.140	.140	.140	.140	.140	.140	.130	.140	.140
LAB-30 (AA)	.123	.129	.132	.130	.138	.141	.132	.136	.139	.132
LAB-31 (AA)	.160	.160	.160	.160	.160	.160	.150	.160	.160	.160
LAB-32 (AA)	.141	.143	.141	.143	.143	.141	.139	.140	.138	.140
LAB-34 (AA)	.145	.146	.144	.146	.144	.147	.146	.146	.146	.146
LAB-35 (AA)	.140	.140	.150	.140	.140	.150	.140	.150	.140	.140
	.150	.150								
LAB-38 (AA)	.154	.154	.165*	.152	.150	.153	.153	.149	.152	.152
LAB-40 (AA)	.140	.145	.146	.148	.147	.145	.140	.144	.146	.146
	.146	.148								
LAB-39 (TITR)	.130	.130	.150	.150	.130					
*LAB-39 (TITR)	.070	.070	.070	.070	.070					
LAB- 1 (COLOR)	.156	.156	.152	.152	.151	.151	.152	.152	.151	.151
LAB- 1 (COLOR)	.145	.144	.145	.142	.147	.146	.142	.147	.147	.145
LAB- 3 (COLOR)	.153	.151	.152	.151	.152	.151	.154	.153	.153	.155

Fe (wt %)

LAB- 1 (AA)	10.91	10.90	10.92	11.06	10.99	11.07	11.07	11.06	11.04	10.97
LAB- 2 (AA)	11.06	11.06	11.06	11.06	11.00	11.26	11.06	11.06	11.14	11.26
LAB-10 (AA)	10.80	10.70	10.74	10.80	10.74	10.70	10.84	10.72	10.82	10.78
LAB-12 (AA)	11.00	11.04	11.00	10.96	10.96	11.04	10.96	11.04	11.00	10.96
LAB-15 (AA)	11.02	10.92	11.02	10.92	11.02	10.92	11.02	11.02	11.12	10.92
*LAB-19 (AA)	12.04	12.02	12.00	12.00	12.05	12.07	12.05	12.04	12.04	12.03
LAB-22 (AA)	10.90	10.90	10.90	10.90	10.90	10.90	10.90	11.00	11.00	11.00
*LAB-32 (AA)	11.48	11.48	11.40	11.48	11.40	11.40	11.33	11.40	11.33	11.40
LAB-38 (AA)	11.32	11.10	11.18	11.11	11.18	11.20	11.19	11.26	11.18	11.30
LAB-40 (AA)	10.65	10.65	10.70	10.85	10.65	10.75	10.80	10.70	10.70	10.85
	10.75	10.85								
LAB- 1 (TITR)	10.85	10.80	10.80	10.80	10.74	10.72	10.74	10.76	10.77	10.81
LAB- 5 (TITR)	11.08	11.11	11.08	11.08	11.08	11.11	11.11	11.11	11.08	11.11
LAB- 6 (TITR)	11.07	11.02	11.03	11.06	11.03	10.99	11.08	11.03	11.00	11.06
LAB- 9 (TITR)	10.71	10.76	10.76	10.76	10.76	10.81	10.81	10.71	10.81	10.71
LAB-13 (TITR)	10.87	10.89	10.89	10.89	10.87	10.89	10.89	10.87	10.87	10.87
LAB-14 (TITR)	11.02	11.02	11.02	11.02	10.97	11.02	10.97	10.97	11.02	11.02
LAB-16 (TITR)	10.94	10.94	10.89	10.86	10.86	10.84	10.89	10.84	10.86	10.76
LAB-18 (TITR)	11.15	11.17	11.20	11.15	11.20	11.12	11.09	11.17	11.22	11.25
	11.28	11.14								
LAB-21 (TITR)	10.91	10.90	10.92	10.91	10.92	10.90	10.90	10.90	10.88	10.94
LAB-23 (TITR)	10.96	10.96	10.96	10.96	10.96	10.92	10.92	10.97	10.92	10.97
LAB-24 (TITR)	10.83	10.81	10.83	10.81	10.82	10.83	10.85	10.83	10.82	10.85
LAB-26 (TITR)	10.70	10.70	10.90	10.80	10.70	10.80	10.80	10.70	10.80	10.80
LAB-29 (TITR)	11.10	11.10	11.20	11.20	11.20	11.10	11.20	11.20	11.10	11.10
LAB-30 (TITR)	10.67	10.57	10.72	10.72	10.67	10.57	10.91	10.72	10.81	10.72
LAB-31 (TITR)	10.91	10.86	10.92	10.91	10.92	10.85	10.93	10.90	10.91	10.96
LAB-34 (TITR)	10.82	10.84	10.82	10.84	10.84	10.88	10.88	10.82	10.86	10.86
LAB-35 (TITR)	10.89	10.92	10.89	10.95						
LAB-39 (TITR)	10.92	10.88	10.98	10.92	10.92					
LAB-39 (TITR)	11.00	11.00	11.02	11.00	11.00					

TABLE 8 (cont'd)

Hg (µg/g)

LAB- 2 (AA)	47	46	45	46	45	45	44	46	45	46
LAB- 5 (AA)	48	50	48	49	48	46	48	46	47	48
LAB- 6 (AA)	40	42	45	44	43	44	44	43	43	44
LAB- 9 (AA)	39	38	38	38	40	39	42	40	40	42
LAB-10 (AA)	46	47	49	44	43	47	48	45	43	44
LAB-14 (AA)	34	37	35	38	35	33	29	33	33	38
LAB-15 (AA)	31	25	23	40	23	34	36	40	36	34
*LAB-18 (AA)	59	59	59	57	58	58	57			
LAB-22 (AA)	47	52	50	49	47	45	45	49	49	48
LAB-23 (AA)	58	57	60	60	56	57	56	51	59	57
LAB-24 (AA)	40	37	44	45	44	41	40	40	39	39
LAB-30 (AA)	42	42	43	43	43	43	42	43	42	43
LAB-35 (AA)	45	46	44	48						
LAB-38 (AA)	44	44	47	46	45	38	38	40	39	42
LAB-40 (AA)	43	41	41	40	41	41	41	41	40	41
LAB-34 (COLOR)	41	41	41	42	40	41	39	40	39	39
*LAB-39 (ES)	50	30	45	60	40					

In (µg/g)

LAB- 5 (AA)	70	70	70	70	70	70	70	70	70	70
LAB- 6 (AA)	75	75	75	72	80	72	80	75	75	77
LAB-10 (AA)	82	80	76	84	82	82	80	80	80	78
LAB-18 (AA)	83	87	85	80						
LAB-34 (AA)	49	51	56	54	54	60	61	59	64	67
LAB-35 (AA)	92	85	86	110	103	80	87	139		
LAB-23 (COLOR)	82	87	86	94	94	92	83	88	83	87
LAB-39 (ES)	130	95	100	115	125					
LAB-40 (ES)	120	110	140	120	130	130	130	150	110	120

MgO (wt %)

LAB- 1 (AA)	.35	.35	.35	.35	.35	.35	.35	.35	.35	.37
LAB- 2 (AA)	.34	.34	.33	.33	.33	.33	.34	.33	.33	.33
LAB- 4 (AA)	.34	.35	.35	.34	.34	.35	.34	.34	.35	.34
LAB- 5 (AA)	.34	.34	.34	.34	.34	.34	.35	.34	.34	.35
LAB- 6 (AA)	.34	.35	.35	.36	.35	.35	.35	.34	.35	.35
LAB- 9 (AA)	.36	.36	.36	.36	.36	.36	.36	.36	.36	.36
LAB-10 (AA)	.30	.30	.30	.30	.30	.30	.30	.30	.30	.30
LAB-12 (AA)	.31	.31	.31	.31	.31	.31	.31	.31	.31	.31
*LAB-13 (AA)	.12	.12	.12	.12	.12	.11	.12	.12	.12	.11
LAB-14 (AA)	.29	.29	.29	.29	.29	.30	.30	.29	.29	.30
*LAB-18 (AA)	.20	.20	.19	.19						
*LAB-19 (AA)	.16	.17	.17	.16	.16	.17	.17	.17	.16	.16
LAB-21 (AA)	.28	.29	.29	.28	.28	.31	.31	.31	.31	.30
LAB-23 (AA)	.29	.30	.29	.29	.29	.29	.29	.30	.29	.29
	.29	.29	.29	.30	.32	.29	.29	.29	.31	.29
LAB-26 (AA)	.30	.30	.30	.30	.29	.30	.30	.29	.30	.29
*LAB-30 (AA)	.12	.11	.11	.11	.11	.11	.11	.11	.11	.11
LAB-34 (AA)	.31	.31	.31	.31	.30	.30	.30	.30	.30	.30
*LAB-35 (AA)	.17	.17	.17	.17						
LAB-38 (AA)	.29	.27	.28	.28	.28	.29	.27	.28	.29	.29

TABLE 8 (cont'd)

Mn (wt %)

LAB- 5 (AA)	.210	.220	.220	.220	.220	.220	.220	.220	.220	.220
LAB- 6 (AA)	.212	.210	.212	.212	.212	.212	.212	.214	.212	.212
LAB- 9 (AA)	.220	.220	.220	.215	.216	.217	.215	.214	.215	.216
LAB-10 (AA)	.211	.207	.206	.205	.205	.210	.205	.206	.211	.205
LAB-14 (AA)	.209	.205	.206	.205	.203	.203	.209	.206	.210	.204
LAB-15 (AA)	.210	.203	.218	.206	.217	.209	.217	.212	.222	.200
LAB-16 (AA)	.232	.232	.232	.236	.240	.229	.231	.231	.228	.226
LAB-18 (AA)	.219	.221	.227	.230	.225					
LAB-19 (AA)	.214	.215	.216	.216	.216	.215	.216	.217	.215	.217
LAB-22 (AA)	.200	.200	.200	.200	.200	.200	.200	.200	.200	.200
LAB-23 (AA)	.240	.230	.230	.230	.230	.230	.230	.230	.230	.230
	.230	.230	.230	.240	.230	.230	.230	.230	.230	.230
LAB-32 (AA)	.230	.229	.230	.230	.231	.229	.231	.229	.229	.229
LAB-34 (AA)	.222	.223	.225	.224	.223	.224	.223	.223	.222	.227
LAB-35 (AA)	.210	.184	.184	.200	.205	.189	.210	.190		
LAB-38 (AA)	.214	.218	.220	.221	.221	.220	.220	.217	.220	.218
LAB-40 (AA)	.240	.220	.221	.225	.231	.230	.240	.220	.230	.223
	.231	.226								
LAB-29 (TITR)	.190	.200	.200	.200	.200	.200	.200	.190	.200	.200
LAB- 3 (COLOR)	.236	.240	.236	.241	.237	.236	.237	.237	.237	.235
LAB-21 (COLOR)	.240	.240	.240	.240	.240	.240	.240	.240	.240	.250
LAB-39 (ES)	.220	.240	.214	.225	.235					

Pb (wt %)

LAB- 1 (AA)	7.25	7.32	7.42	7.35	7.28	7.33	7.40	7.24	7.33	7.37
LAB- 2 (AA)	7.22	7.24	7.34	7.28	7.34	7.32	7.28	7.32	7.32	7.22
LAB- 6 (AA)	7.50	7.44	7.43	7.53	7.44	7.43	7.44	7.53	7.43	7.42
LAB- 9 (AA)	7.30	7.40	7.40	7.20	7.20	7.00	7.60	7.50	7.40	7.60
LAB-10 (AA)	7.45	7.46	7.46	7.47	7.46	7.46	7.49	7.45	7.45	7.46
LAB-15 (AA)	7.40	7.43	7.44	7.41	7.51	7.42	7.49	7.50	7.53	7.43
LAB-16 (AA)	7.72	7.72	7.72	7.70	7.70	7.67	7.72	7.82	7.76	7.74
*LAB-19 (AA)	6.83	6.85	6.85	6.78	6.80	6.80	6.75	6.78	6.80	6.80
LAB-21 (AA)	7.59	7.59	7.59	7.59	7.59	7.42	7.43	7.45	7.38	7.42
LAB-22 (AA)	7.54	7.50	7.49	7.45	7.45	7.49	7.49	7.50	7.50	7.50
LAB-23 (AA)	7.50	7.50	7.50	7.50	7.40	7.50	7.50	7.50	7.40	7.40
LAB-26 (AA)	7.55	7.40	7.35	7.65	7.40	7.58	7.50	7.50	7.75	7.40
LAB-31 (AA)	7.33	7.34	7.43	7.36	7.34	7.33	7.34	7.30	7.34	7.34
LAB-32 (AA)	7.38	7.41	7.32	7.47	7.41	7.42	7.41	7.41	7.42	7.52
LAB-34 (AA)	7.42	7.38	7.39	7.47	7.48	7.37	7.41	7.42	7.39	7.45
LAB-38 (AA)	7.68	7.70	7.69	7.76	7.79	7.67	7.73	7.67	7.67	7.68
LAB-40 (AA)	7.10	7.58	7.80	7.55	7.65	7.70	7.15	7.55	7.80	7.55
	7.75	7.75								
LAB- 1 (TITR)	7.45	7.45	7.48	7.43	7.43	7.48	7.39	7.41	7.40	7.43
LAB- 3 (TITR)	7.44	7.43	7.42	7.44	7.42	7.39	7.37	7.38	7.35	7.37
LAB- 5 (TITR)	7.61	7.62	7.57	7.60	7.63	7.54	7.60	7.56	7.62	7.56
LAB-13 (TITR)	7.69	7.69	7.69	7.69	7.67	7.69	7.69	7.67	7.67	7.67
LAB-14 (TITR)	7.49	7.44	7.44	7.55	7.49	7.49	7.49	7.44	7.49	7.44
LAB-16 (TITR)	7.42	7.52	7.54	7.52	7.53	7.48	7.49	7.50	7.50	7.46
LAB-18 (TITR)	7.19	7.27	7.33	7.37	7.34	7.25	7.20	7.40	7.31	7.33
	7.25	7.28								
LAB-24 (TITR)	7.47	7.44	7.47	7.44	7.44	7.47	7.47	7.44	7.41	7.44
LAB-30 (TITR)	7.32	7.37	7.37	7.32	7.37	7.37	7.42	7.32	7.37	7.32
LAB-33 (TITR)	7.45	7.35	7.40	7.35	7.37	7.53	7.40	7.45	7.30	7.37
LAB-34 (TITR)	7.43	7.41	7.43	7.46	7.41	7.41	7.46	7.41	7.48	7.48
LAB-35 (TITR)	7.37	7.43	7.38	7.46	7.37	7.44	7.41	7.46		
LAB-45 (TITR)	7.44	7.45	7.44	7.46	7.46	7.45	7.47	7.45	7.46	7.43
LAB-29 (GRAV)	7.30	7.25	7.30	7.25	7.21	7.23	7.33	7.28	7.31	7.28
LAB-39 (GRAV)	6.88	7.38	7.05	6.98	6.88					
*LAB-39 (GRAV)	7.05	7.01	6.46	6.73	6.92					
*LAB-38 (XRF)	7.95	7.90	7.94	7.95	7.98	7.81	7.90	7.87	7.93	7.94

TABLE 8 (cont'd)

S (wt %)

LAB- 5 (GRAV)	30.38	30.48	30.33	30.40	30.40	30.35	30.46	30.37	30.45	30.35
LAB- 6 (GRAV)	29.79	29.57	29.72	29.76	29.86	29.35	29.47	30.00	29.34	29.96
LAB- 9 (GRAV)	29.59	29.56	29.65	29.54	29.56	29.51	29.67	29.67	29.67	29.56
LAB-10 (GRAV)	30.20	30.10	30.26	30.20	30.28	30.30	30.15	30.20	30.20	30.18
LAB-13 (GRAV)	30.33	30.28	30.31	30.31	30.36	30.45	30.38	30.37	30.41	30.36
LAB-14 (GRAV)	30.37	30.43	30.31	30.12	30.28	30.43	30.15	30.25	30.21	30.49
LAB-15 (GRAV)	30.16	30.66	30.96	30.66	30.66	30.46	31.16	30.76	30.96	30.96
LAB-18 (GRAV)	30.24	30.28	30.15	30.40	30.25	30.12	30.19	30.24	30.09	30.14
	30.28	30.17								
LAB-21 (GRAV)	30.03	30.02	30.05	30.17	30.16	30.24	30.25	30.23	30.23	30.37
LAB-23 (GRAV)	29.77	29.90	29.94	30.04	29.99	29.78	29.91	29.85	29.90	
*LAB-30 (GRAV)	27.80	28.20	27.50	27.90	28.10	29.10	29.10	28.00	29.10	28.20
LAB-31 (GRAV)	29.57	29.66	29.73	29.68	29.70	29.70	29.56	29.53	29.60	29.53
LAB-34 (GRAV)	30.61	30.67	30.78	30.70	30.81	30.72	30.72	30.80	30.23	31.16
LAB-35 (GRAV)	30.69	30.69	30.40	30.52						
LAB-38 (GRAV)	30.11	30.12	30.11	30.21	30.06	30.29	30.11	30.08	30.34	30.18
LAB-39 (GRAV)	31.08	30.83	31.95*	31.02	31.12					
*LAB-39 (GRAV)	32.81	31.41	31.95	31.65	31.84					
*LAB-14 (COMB)	31.25	31.40	31.40	31.60	31.85	31.65	31.90	31.25	31.30	31.70
*LAB-26 (COMB)	32.20	32.00	32.20	31.80	30.90	31.40	31.80	32.20	32.40	31.40

Sb (wt %)

LAB- 5 (AA)	.047	.047	.047	.049	.047	.047	.047	.047	.048	.048
LAB- 6 (AA)	.054	.055	.055	.055	.054	.055	.056	.053	.056	.053
LAB- 9 (AA)	.052	.054	.058	.058	.056	.056	.064	.064	.060	.062
LAB-10 (AA)	.053	.051	.052	.050	.053	.052	.052	.051	.051	.052
LAB-14 (AA)	.059	.059	.057	.057	.057	.056	.058	.058	.056	.057
*LAB-18 (AA)	.020	.020	.020	.020						
*LAB-19 (AA)	.037	.039	.037	.039	.036	.037	.035	.036	.037	.037
LAB-21 (AA)	.059	.058	.059	.060	.059	.059	.059	.059	.059	.060
LAB-24 (AA)	.052	.053	.048	.046	.046	.052	.048	.052	.050	.054
LAB-30 (AA)	.051	.055	.055	.053	.054	.053	.054	.054	.051	.055
LAB-34 (AA)	.048	.048	.048	.050	.048	.047	.048	.050	.047	.049
LAB-34 (AA)	.047	.050	.044	.047	.046	.046	.050	.049	.045	.051
LAB-35 (AA)	.049	.052	.057	.050	.055	.055	.048	.051	.054	.049
	.057	.052								
LAB- 3 (COLOR)	.053	.054	.055	.054	.054	.054	.054	.054	.055	.054
LAB-23 (COLOR)	.053	.050	.055	.055	.053	.053	.052	.055	.053	.055
*LAB-39 (ES)	.035	.038	.027	.029	.032					
LAB-40 (ES)	.042	.042	.050	.044	.051	.042	.044	.050	.049	.050

Se (µg/g)

LAB-15 (AA)	5	5	5	5	6	6	5	6	6	6
LAB-18 (AA)	3	6	6	5	5	5	6	7		
LAB-24 (AA)	5	5	5	5	5	5	5	6	5	5
LAB-34 (AA)	3	3	4	3	4	3	3	3	3	3
LAB- 1 (COLOR)	8	8	7	8	8	6	6	6	5	6
LAB- 3 (COLOR)	5	6	5	5	5	5	5	5	5	5
LAB- 9 (COLOR)	4	4	5	4	4	5	4	5	5	5
LAB-10 (COLOR)	4	4	4	4	4	4	4	4	4	4
LAB-23 (COLOR)	6	6	6	6	6	6	6	6	6	6

TABLE 8 (cont'd)

SiO_2 (wt %)

LAB- 2 (AA)	1.12	1.10	1.07	1.06	1.05	1.04	1.05	1.09	1.09	1.11
LAB-10 (AA)	.99	.98	.99	.99	.98	1.00	.99	.99	.99	.98
LAB-15 (AA)	.96	.91	.91	.96	.91	.91	.91	.91	.91	.96
LAB-26 (AA)	1.09	1.00	1.00	1.17	1.00	1.00	1.00	1.09	1.00	1.00
LAB- 1 (GRAV)	1.03	1.05	1.03	1.05	1.01	.98	1.03	.98	1.03	.98
LAB- 4 (GRAV)	1.07	1.06	1.05	1.06	1.05	1.07	1.05	1.05	1.05	1.04
LAB- 5 (GRAV)	.66	.66	.65	.68	.69	.65	.66	.68	.66	.65
LAB- 9 (GRAV)	1.35	1.35	1.35	1.35	1.35	1.25	1.25	1.25	1.25	1.25
LAB-13 (GRAV)	.69	.71	.73	.72	.69	.73	.73	.69	.72	.73
LAB-14 (GRAV)	1.13	1.10	1.13	1.06	1.07	1.11	1.07	1.05	1.34	1.29
LAB-18 (GRAV)	1.00	1.04	1.01	.98	1.00	1.00	.98	1.04		
LAB-19 (GRAV)	1.06	1.08	1.09	1.06	1.09	1.08	1.09	1.05	1.10	1.08
LAB-21 (GRAV)	.76	.76	.76	.74	.71	.73	.70	.72	.73	.72
LAB-23 (GRAV)	1.11	1.11	1.13	1.12	1.09	1.11	1.09	1.08	1.09	1.10
LAB-24 (GRAV)	.97	.96	.96	.95	.97	.96	.95	.96	.97	.97
LAB-29 (GRAV)	1.18	1.16	1.20	1.16	1.17	1.14	1.26	1.26	1.16	1.26
LAB-34 (GRAV)	1.02	1.01	1.01	1.00	1.03	1.01	1.00	.99	.99	.99
LAB-35 (GRAV)	.92	.81	.99	.82	.95	.82	.98	.82		
LAB-39 (GRAV)	1.02	1.06	.98	.98	1.10					
LAB-39 (GRAV)	1.29	1.45	1.31	1.34	1.27					

Sn (μg/g)

LAB-23 (AA)	100	80	80	80	80	100	100	80	100	100
	80	50*	100	100	100	100	100	100	100	80
LAB- 1 (COLOR)	75	75	76	76	74	76	76	89	77	81
LAB-34 (COLOR)	49	53	48	51	49	50	51	49	48	51
*LAB- 9 (ES)	200	260	220	200	240	200	190	200	180	200
LAB-18 (ES)	20	20	20	20						
LAB-39 (ES)	105	80	110	85	90					
LAB-40 (ES)	85	75	68	64	67	72	80	67	67	64
LAB-10 (POLAR)	72	70	80	72	72	69	72	72	71	70

Te (μg/g)

LAB-34 (AA)	.3	.2	.2	.3	.3	.2	.3	.2	.2	.2

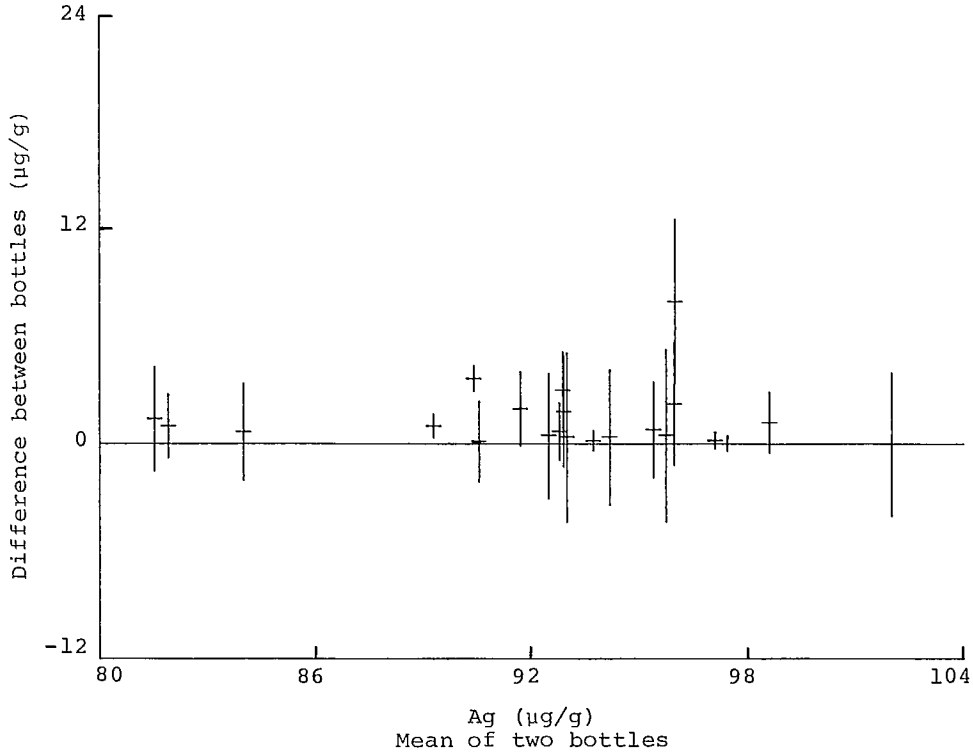
TABLE 8 (cont'd)

	Zn (wt %)									
LAB- 2 (AA)	44.72	44.72	45.00	44.72	44.86	44.86	44.86	44.72	44.60	44.86
LAB-15 (AA)	44.19	44.79	44.59	44.49	44.39	45.09	44.29	44.79	44.29	44.49
LAB-19 (AA)	45.00	45.63	45.50	45.00	44.75	45.00	45.25	45.50	45.50	45.25
LAB-26 (AA)	44.20	44.60	44.20	44.00	44.40	44.20	44.00	44.20	44.60	44.00
LAB-32 (AA)	45.38	45.45	45.33	45.20	45.25	45.00	45.33	45.33	45.00	45.20
LAB- 1 (TITR)	44.80	44.73	44.76	44.95	44.81	44.85	44.79	44.78	44.81	44.78
	44.60	44.81	44.76	44.69	44.73	44.73	44.69	44.81	44.86	44.77
	44.71	44.81	44.59	44.94	44.75					
LAB- 4 (TITR)	44.79	44.85	44.82	44.84	44.82	44.75	44.72	44.90	44.91	44.94
LAB- 5 (TITR)	44.58	44.51	44.56	44.54	44.57	44.54	44.55	44.59	44.63	44.56
LAB- 6 (TITR)	44.60	44.42	44.45	44.36	44.47	44.46	44.66	44.44	44.53	44.58
LAB- 6 (TITR)	44.78	44.54	44.52	44.62	44.70	44.64	44.59	44.70	44.66	44.71
LAB- 9 (TITR)	44.31	44.35	44.40	44.31	44.31	44.35	44.33	44.31	44.35	44.31
LAB-13 (TITR)	45.30	45.30	45.20	45.20	45.20	45.30	45.09	45.20	45.20	45.20
LAB-14 (TITR)	44.50	44.50	44.40	44.40	44.50	44.50	44.40	44.50	44.50	44.60
*LAB-16 (TITR)	44.73	44.81	44.83	44.66	44.93	44.96	44.90	44.52	44.71	44.76
LAB-16 (TITR)	44.89	45.02	45.00	45.07	45.11	44.83	44.88	44.84	44.96	44.98
LAB-16 (TITR)	45.42	45.58	45.55	45.57	45.44	45.48	45.44	45.49		
LAB-18 (TITR)	45.12	45.22	45.22	45.20	45.22	45.25	45.35	45.20	45.20	45.20
	45.12	45.24								
LAB-21 (TITR)	44.64	44.57	44.68	44.60	44.68	44.64	44.60	44.59	44.67	44.67
LAB-22 (TITR)	44.70	44.60	44.50	44.60	44.60	44.50	44.50	44.60	44.60	44.60
LAB-23 (TITR)	45.00	45.10	45.00	44.90	45.10	45.10	45.00	44.90	45.10	45.10
LAB-24 (TITR)	45.02	45.03	45.02	44.81	45.04	45.03	45.01	45.04	44.81	45.01
LAB-24 (TITR)	44.54	44.51	44.68	44.42	44.60	44.52	44.54	44.80	44.55	44.51
LAB-29 (TITR)	44.80	44.70	44.70	44.80	44.90	44.80	44.80	44.80	44.80	44.80
LAB-30 (TITR)	44.82	45.01	44.67	44.74	44.88	44.78	44.68	44.86	44.76	44.74
LAB-31 (TITR)	45.04	45.36	45.03	45.29	45.19	45.25	45.51	45.53	45.34	
LAB-33 (TITR)	44.88	44.68	44.88	44.88	44.78	44.85	44.95			
LAB-33 (TITR)	44.84	44.82	44.68							
LAB-34 (TITR)	44.49	44.59	44.59	44.52	44.54	44.63	44.67	44.63	44.59	44.66
LAB-35 (TITR)	44.84	44.98	44.70	44.77	44.79	44.88	44.85	44.98	44.70	44.70
	44.80	44.90								
LAB-37 (TITR)	44.86	44.95	45.05	45.02	45.02	44.87	44.80	45.09	45.07	45.02
LAB-38 (TITR)	44.70	44.58	44.43	44.56	44.52	44.38	44.35	44.40	44.42	44.24
LAB-39 (TITR)	44.14	44.40	44.60	44.25	44.60					
LAB-39 (TITR)	45.50	45.50	44.80	45.50	44.80					
LAB-40 (TITR)	44.60	44.40	44.90	44.30	44.70	44.50	44.70	44.70	44.60	44.90
	44.70	44.60	44.50	44.50						
LAB-45 (TITR)	44.28	44.38	44.27	44.30	44.40	44.36	44.35	44.35	44.32	44.33
LAB-45 (TITR)	44.32	44.37	44.33	44.27	44.33	44.39	44.39	44.29	44.30	44.35
LAB-10 (POLAR)	44.50	44.52	44.40	44.52	44.50	44.54	44.50	44.50	44.46	44.38

* 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 - titrimetrv; COLOR - colorimetry (spectrophotometry); GRAV - gravimetry; ES - emission spectroscopy; FA - fire assay with gravimetric finish; FA-AA - fire assay with atomic absorption finish; POLAR - polarography; NAA - neutron activation analysis; COMB - combustion; XRF - X-ray fluorescence.



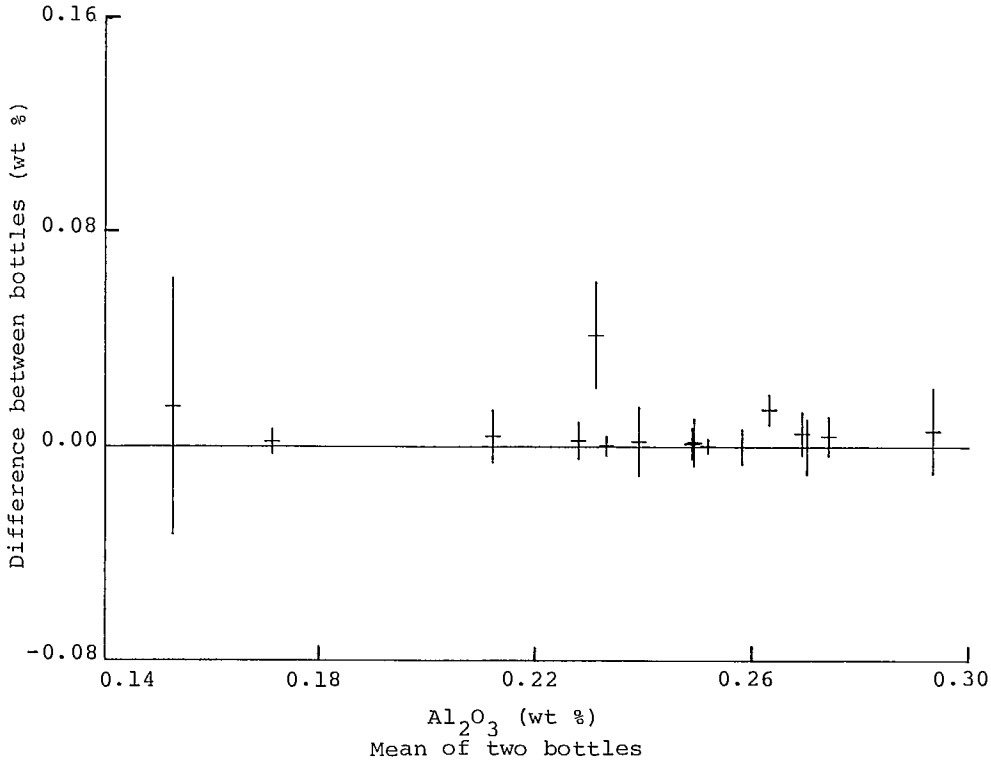
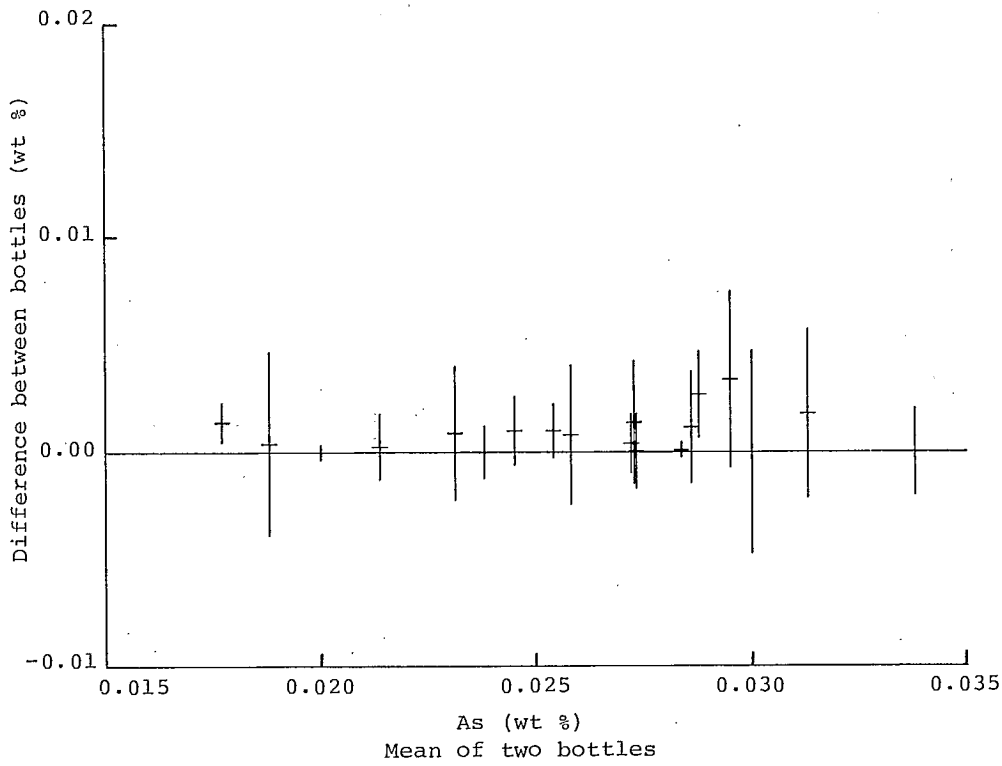


Fig. 1 - Degree of homogeneity of CZN-1. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.


0.02
0.01
0.00
-0.01
Difference between bottles (wt %)
0.015
0.020
0.025
0.030
0.035
As (wt %)
Mean of two bottles

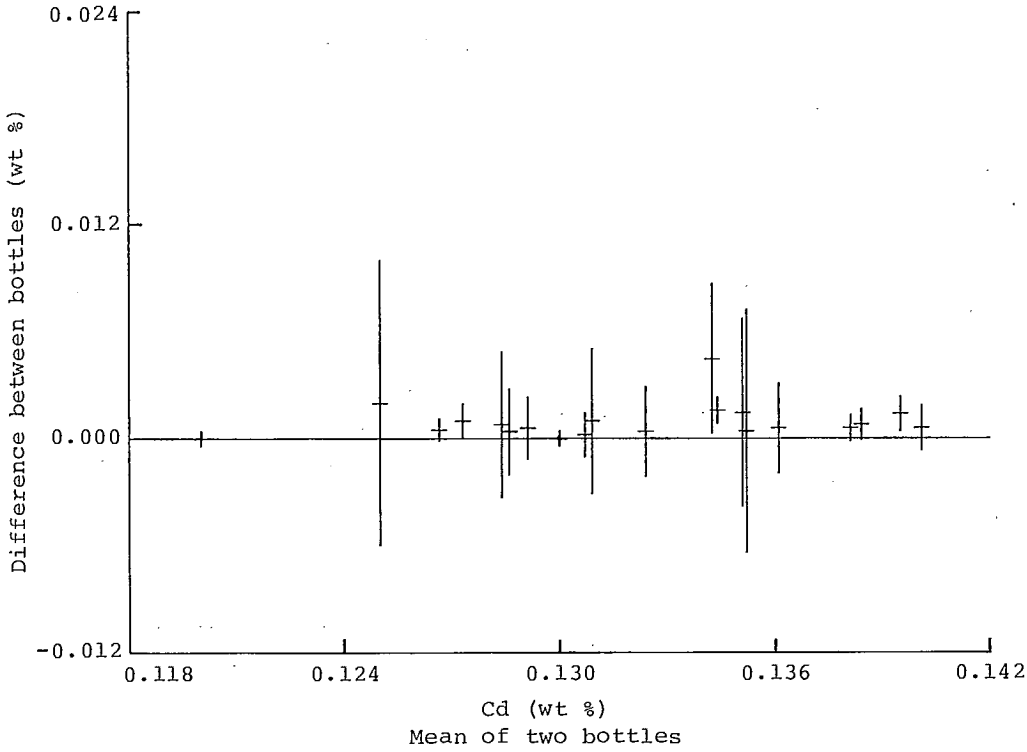
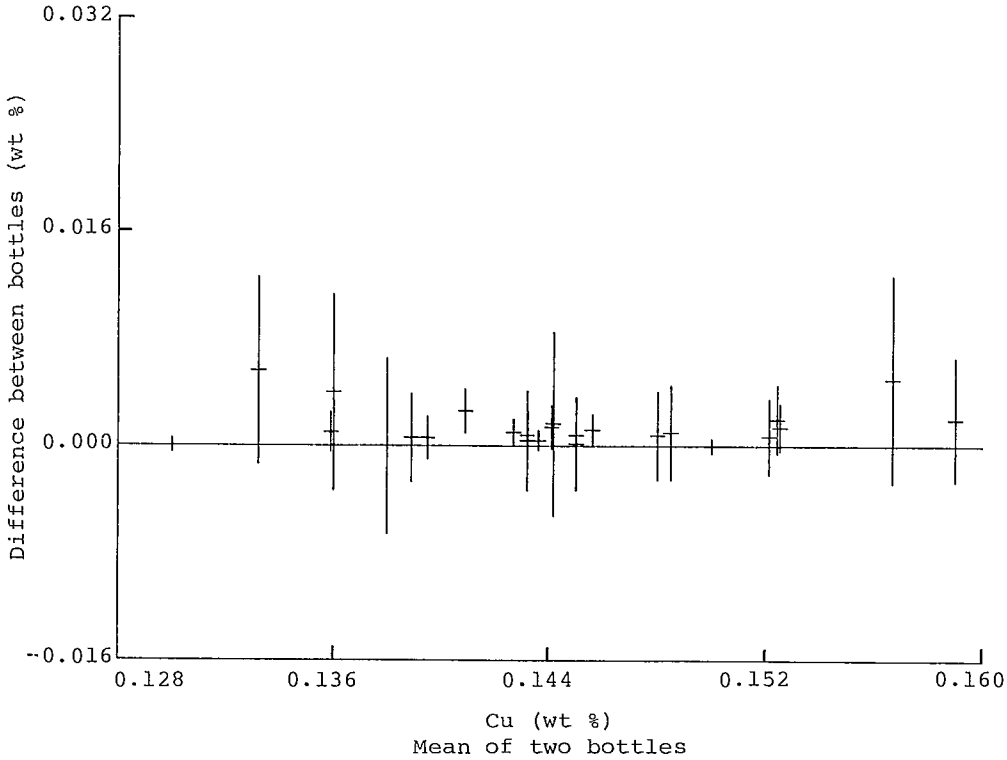

0.024
0.012
0.000
-0.012
Difference between bottles (wt %)
0.118
0.124
0.130
0.136
0.142
Cd (wt %)
Mean of two bottles

Fig. 1 (cont'd)


0.032
0.016
0.000
-0.016
Difference between bottles (wt %)
0.128
0.136
0.144
0.152
0.160
Cu (wt %)
Mean of two bottles

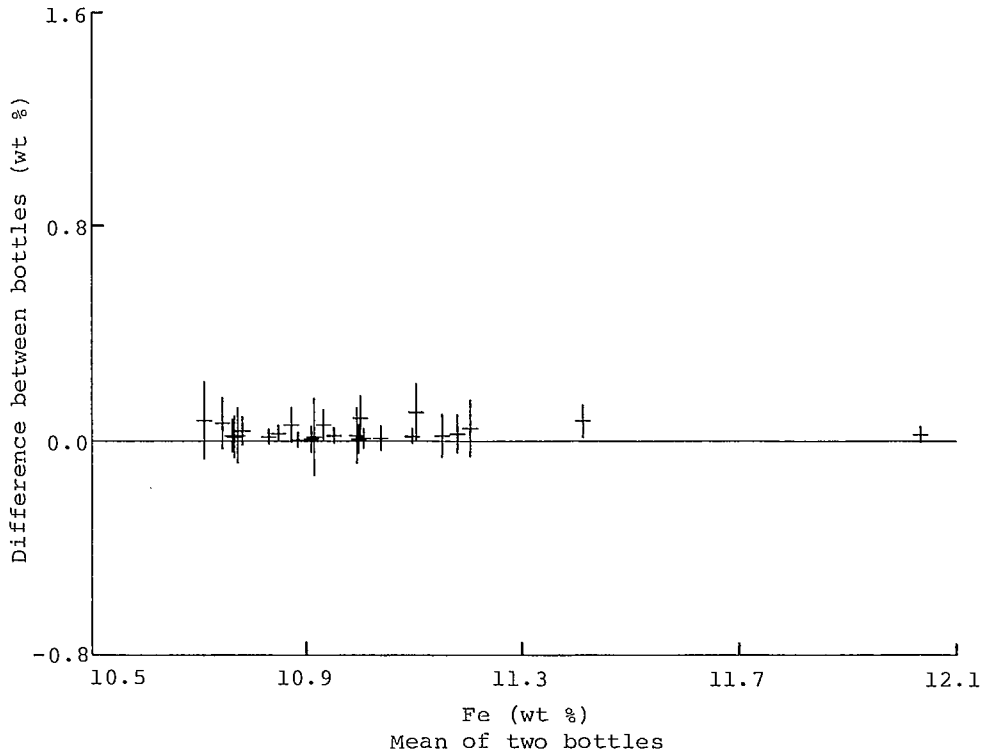
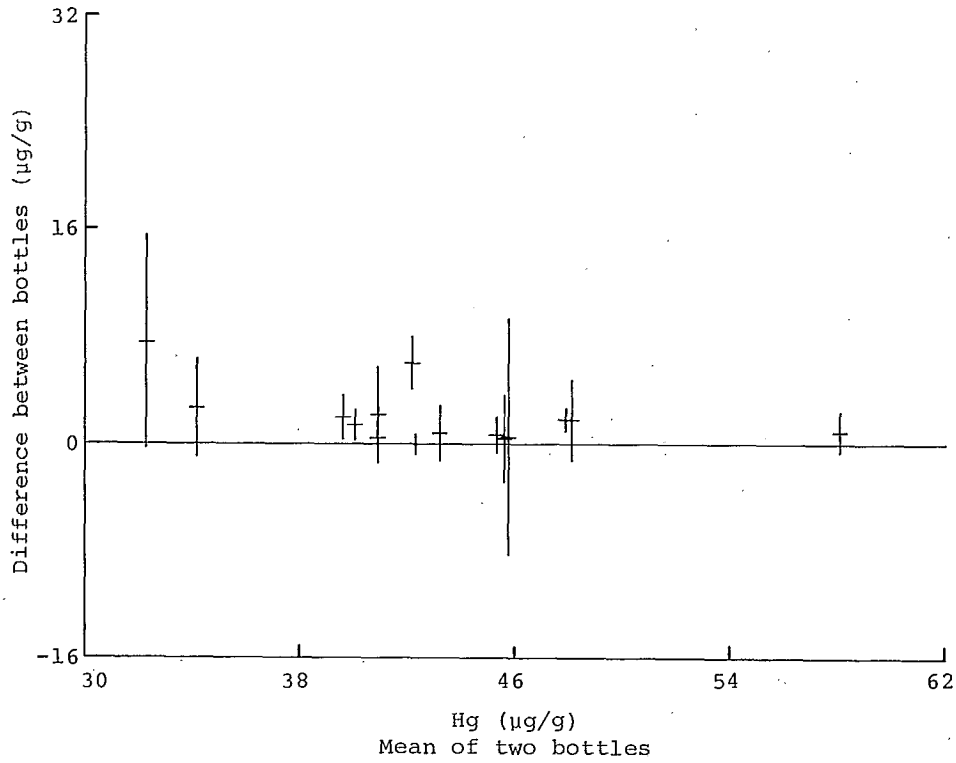

1.6
0.8
0.0
-0.8
Difference between bottles (wt %)
10.5
10.9
11.3
11.7
12.1
Fe (wt %)
Mean of two bottles

Fig. 1 (cont'd)


32
16
0
-16
Difference between bottles (µg/g)
30
38
46
54
62
Hg (µg/g)
Mean of two bottles

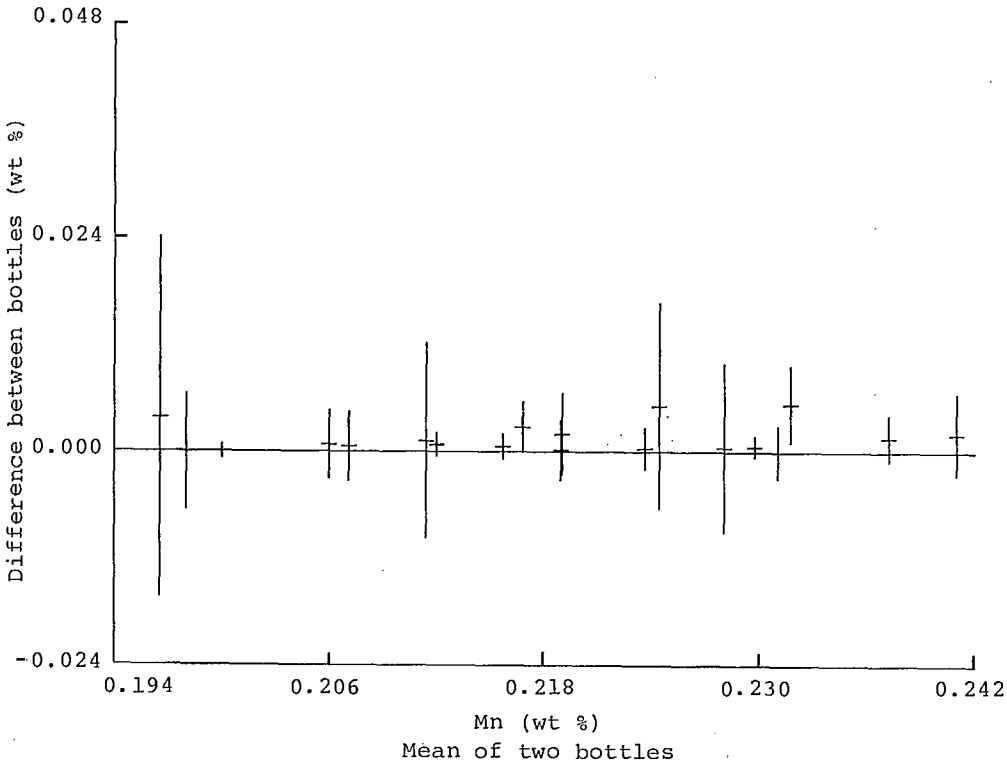
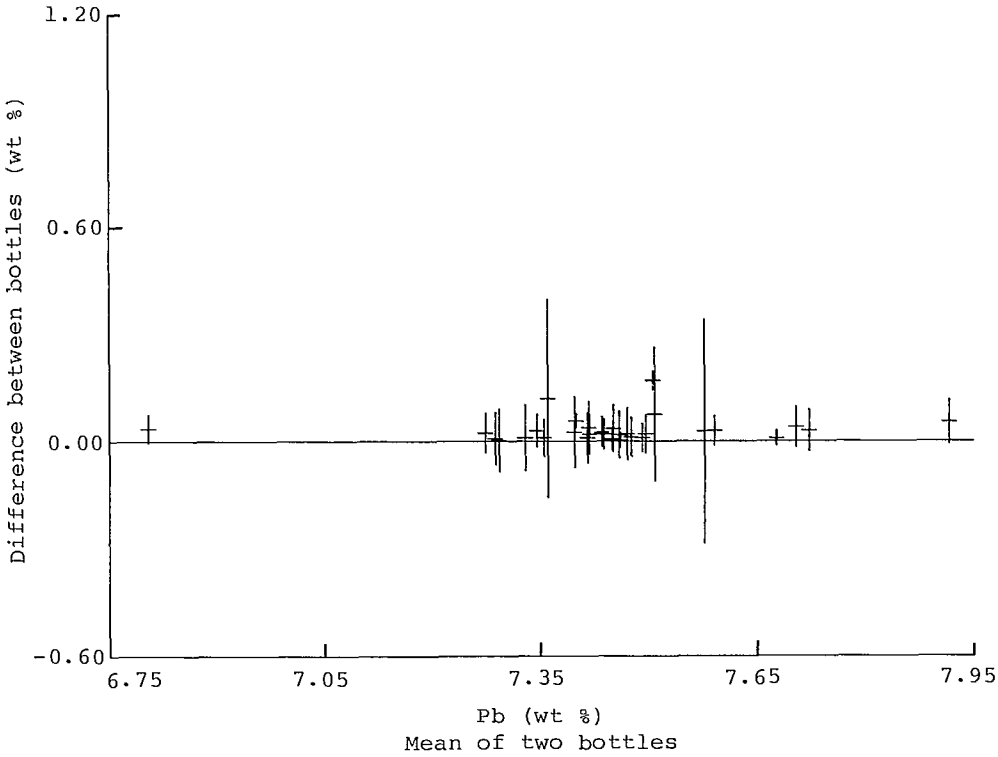

0.048
0.024
0.000
-0.024
Difference between bottles (wt %)
0.194
0.206
0.218
0.230
0.242
Mn (wt %)
Mean of two bottles

Fig. 1 (cont'd)


1.20
0.60
0.00
-0.60
Difference between bottles (wt %)
6.75
7.05
7.35
7.65
7.95
Pb (wt %)
Mean of two bottles

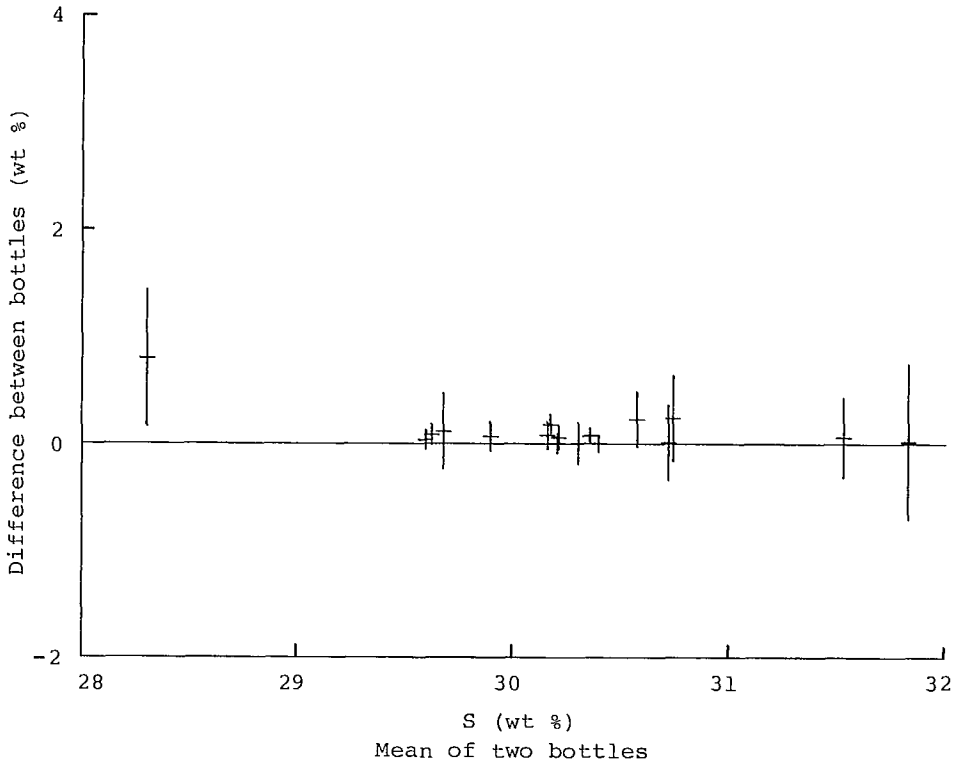
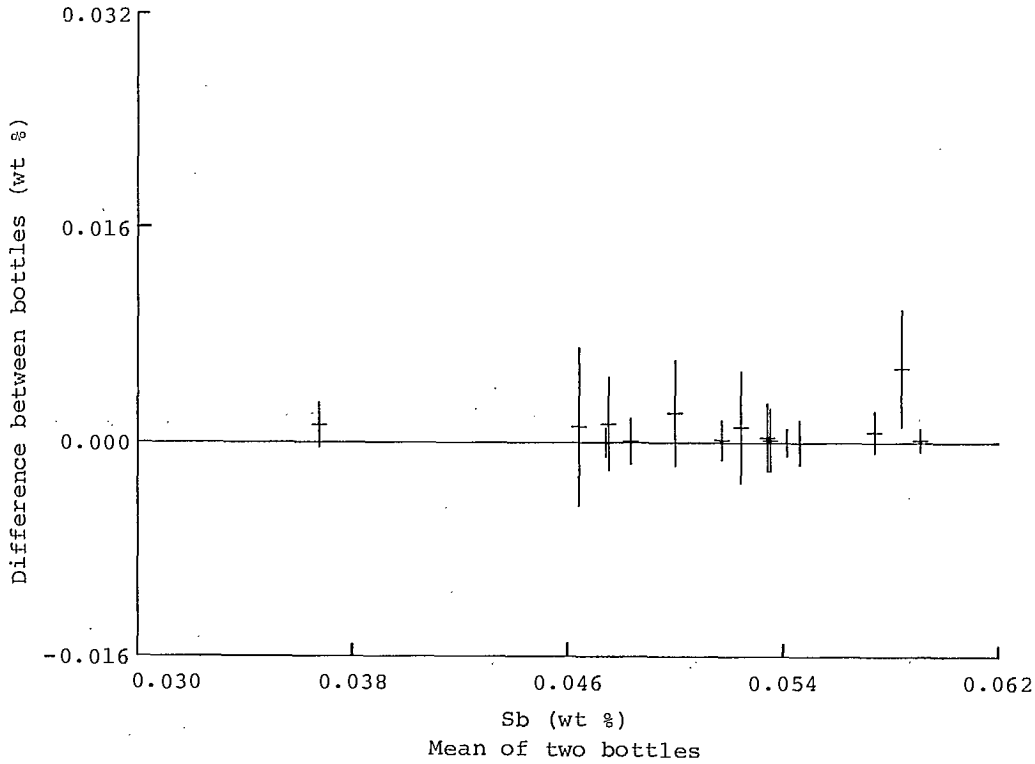

4
2
0
-2
Difference between bottles (wt %)
28
29
30
31
32
S (wt %)
Mean of two bottles

Fig. 1 (cont'd)


0.032
0.016
0.000
-0.016
Difference between bottles (wt %)
0.030
0.038
0.046
0.054
0.062
Sb (wt %)
Mean of two bottles

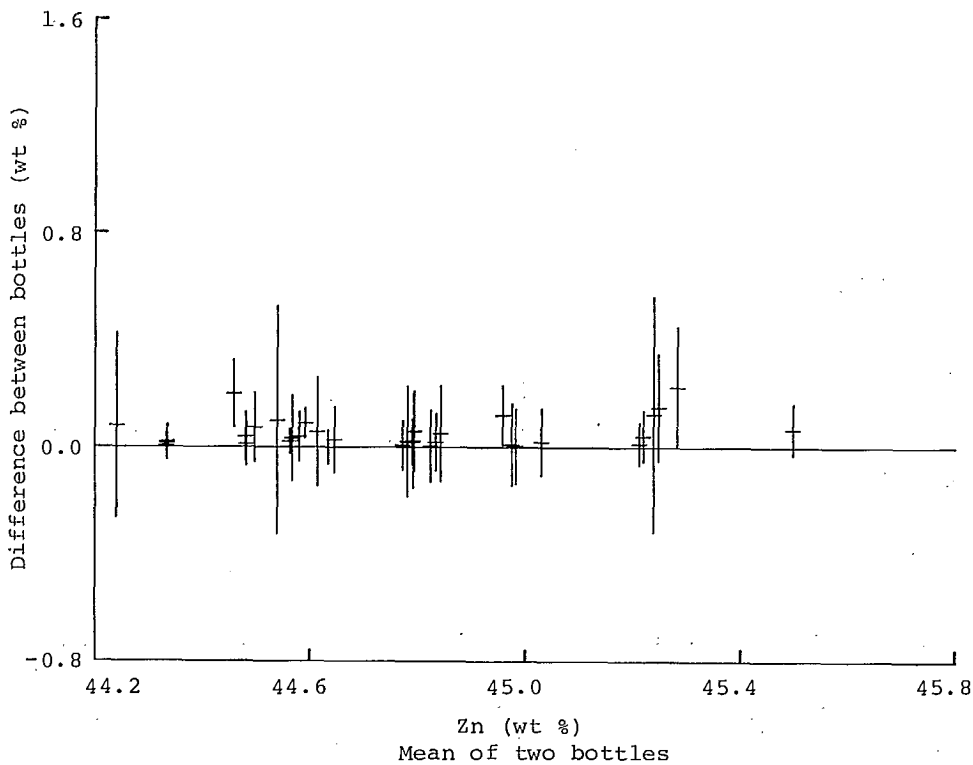

1.6
0.8
0.0
-0.8
Difference between bottles (wt %)
44.2
44.6
45.0
45.4
45.8
Zn (wt %)
Mean of two bottles

Fig. 1 (cont'd)

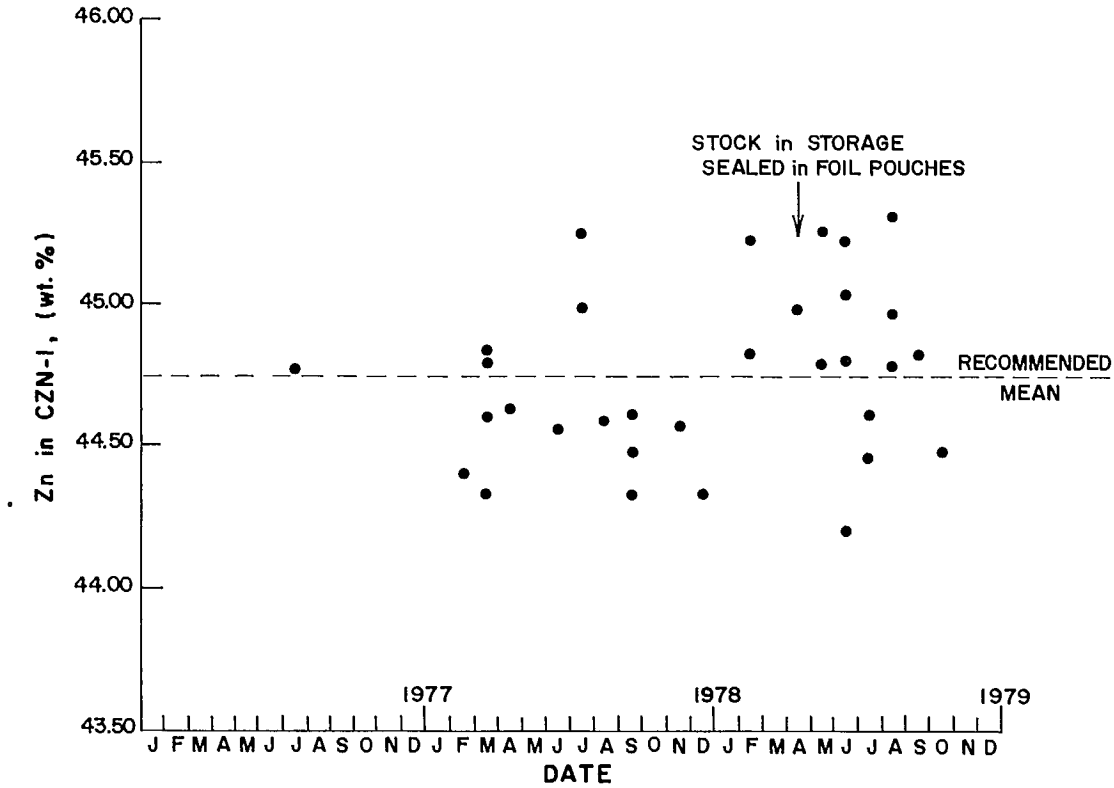


Fig. 2. - Laboratory means for Zn vs date of analysis

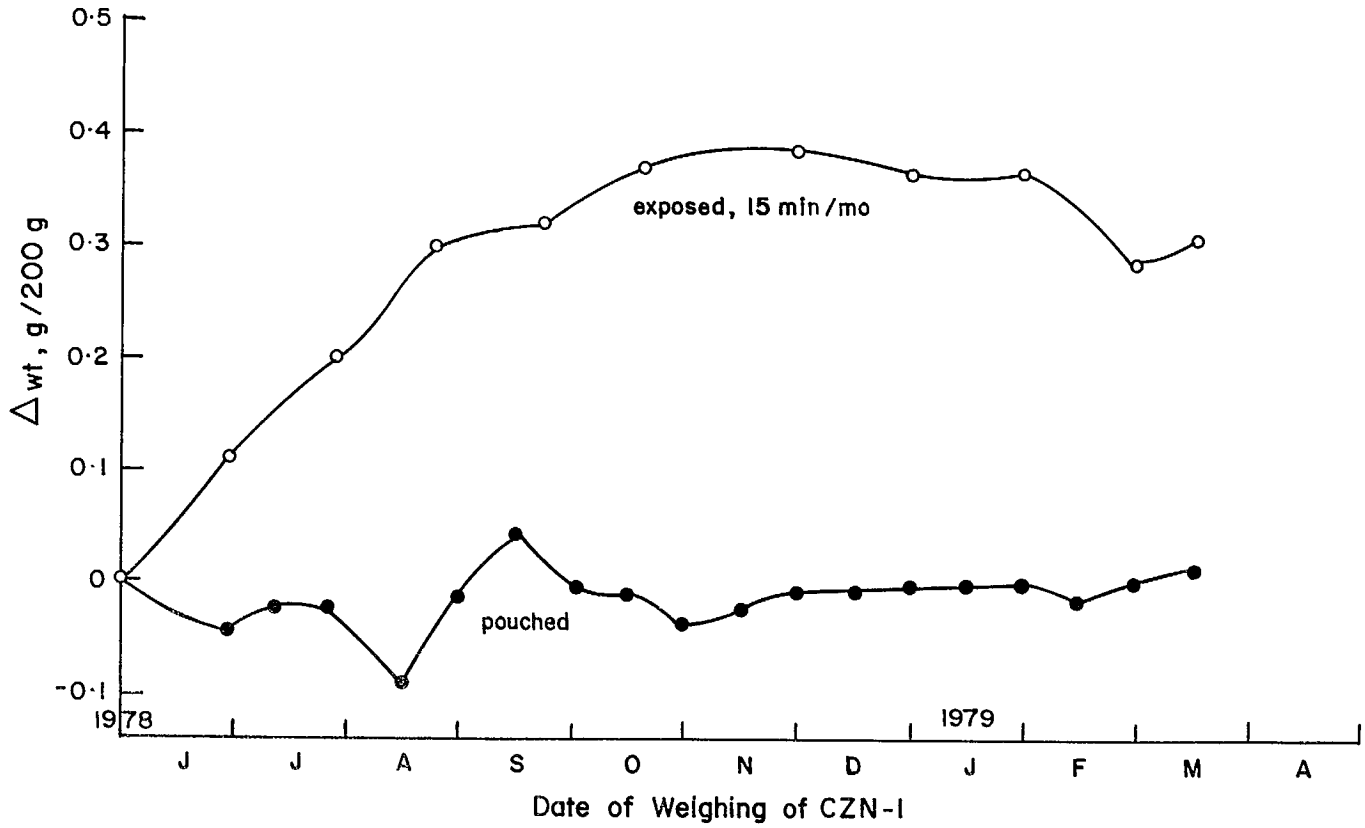


Fig. 3. - Change in weight of pouched and unpouched samples of CZN-1 vs storage time

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.

CANMET, Energy, Mines and Resources Canada, Mineral Sciences Laboratories, Ottawa, Ontario (nine 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.

Falconbridge Copper Ltd., Sturgeon Lake Joint Venture, Ignace, Ontario.

Falconbridge Nickel Mines Ltd., Metallurgical Laboratories, Thornhill, Ontario.

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.

Lakefield Research of Canada Ltd., Lakefield, Ontario.

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.

Sherritt Gordon Mines Ltd., Research and Development Division, Fort Saskatchewan, Alberta.

Sherritt Gordon Mines Ltd., Mining Division, Lynn Lake, Manitoba.

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 ZINC IN CZN-1

The titrimetric methods for zinc outlined below were used by a relatively large proportion of contributing laboratories and they cannot 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.

EDTA Method (1A) (Laboratories 1, 4, 6, 18, 22, 24, 34 and 38)

After sample decomposition and removal of lead as the sulphate, zinc was separated by MIBK extraction of its thiocyanate complex from a 0.8 mol/L ammonium thiocyanate-dilute acid medium containing thiourea and ammonium fluoride as complexing agents for interfering elements. Zinc was leach-extracted with ammonium chloride-ammonium hydroxide solutions and liberated from its cyanide complex with formaldehyde. At pH 10, in the presence of ascorbic acid and sodium cyanide, zinc was titrated with EDTA using Eriochrome Black-T as internal indicator.

Ferrocyanide Method (Laboratories 6, 9, 21, 23, 30 and 37)

After sample decomposition, iron and manganese were removed from the solution by repeated treatment with ammonium hydroxide-ammonium chloride solution containing persulphate. The acidity of the filtrate was adjusted with hydrochloric acid and the sample solution was boiled with test lead to remove copper. Zinc was titrated at approximately 70°C with ferrocyanide solution using ammonium molybdate or uranyl acetate as an external indicator.

REFERENCE

1A. Kinnunen, J. and Wennerstrand, B. "Rapid EDTA titration of zinc following thiocyanate extraction"; Chemist Analyst; 42:80-83; 1953.