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COPPER CONCENTRATE CCU-1 — A CERTIFIED REFERENCE MATERIAL

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

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COPPER CONCENTRATE CCU-1 - A CERTIFIED REFERENCE MATERIAL

by

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

SYNOPSIS

A 200-kg sample of a copper (flotation) concentrate, CCU-1, from Lynn Lake, Manitoba has been prepared as a compositional reference material. CCU-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, 33 laboratories contributed results for one or more of 18 elements in each of two bottles of CCU-1. Based on a statistical analysis of the data, recommended values have been assigned for: Ag, Al_2O_3, Au, Cu, Hg, Pb, SiO_2 and Zn. Also, non-certified values have been determined for: As, Bi, CaO, Cd, Fe, MgO, Mo, S, Se, and Te.

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Note: Major contributions to the certification of CCU-1 were also made by other staff members of the Mineral Sciences Laboratories as well as by those of laboratories in other organizations.

CONCENTRE DE CUIVRE CCU-1 - UN MATERIAU DE REFERENCE CERTIFIE

par

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

SOMMAIRE

Un échantillon de 200 kg de concentré de cuivre (flottation), CCU-1, provenant de Lynn Lake au Manitoba, a été préparé en tant que matériau de référence de composition. Le CCU-1 a été broyé jusqu'à -74 µm et mélangé ensemble; l'homogénéité a été vérifiée par des méthodes chimiques et radiographiques et ce matériau a ensuite été embouteillé en contenants de 200 g.

Trente-trois laboratoires ont fourni des résultats pour un ou plusieurs des 18 éléments dans chacune des deux bouteilles de CCU-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, Au, Cu, Hg, Pb, SiO_2 et Zn. Aussi des valeurs non-homologuées ont été determinées pour l'As, Bi, CaO, Cd, Fe, MgO, Mo, S 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 copper concentrate CCU-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 sciences laboratories. Certified reference materials issued previously by CCRMP are described in a catalogue available from CANMET, Energy, Mines and Resources Canada, Ottawa (1).

CCU-1 was chosen as a reference material because it contains a relatively large number of minor and trace elements at useful levels of concentration. It was donated to CCRMP in late 1975 by Sherritt Gordon Mines Ltd. and was a flotation concentrate of ore from the Ruttan Mine at Lynn Lake, Manitoba. The 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 18 elements by 33 laboratories which used methods of their choice. Recommended values for the eight certified elements are given in Table 3; methodological, statistical and other analytical information is presented in Tables 4 to 8.

TABLE 1

Approximate mineralogical composition of CCU-1

Mineral	wt %
Chalcopyrite	82
Pyrite	9
Sphalerite	9
Pyrrhotite	trace

TABLE 2

Particle size analysis (wet screen)

Size of fraction (μm)	wt %
-104 + 74	1
-74 + 55	13
-55 + 46	12
-46 + 37	2
-37	72

PREPARATION OF CCU-1

In early 1976, CCU-1, which had been dried at ~100°C, was ground to pass a 74-μm screen. The powdered concentrate, weighing approximately 200 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.

INTERLABORATORY PROGRAM FOR CERTIFICATION OF CCU-1

The laboratories that participated in the certification program for CCU-1 are listed alphabetically in Appendix A. Each was arbitrarily assigned a code number so that analytical results could be recorded while preserving anonymity. 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 mode 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 CCU-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 treatment 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 8 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 CCU-1 (Table 7)*. The degree of homogeneity of CCU-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):

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

* For certain critical applications the between-bottle variance may have to be taken into account.

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 means 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.

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

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

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 8 certified constituents of CCU-1 are given in Table 3 along with the consensus values which are boxed in for easy identification.

Similar statistics for 10 non certified constituents are given in Table 4.

Discussion of analytical results

An outline of the principal titrimetric methods used for copper in CCU-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 the subsample 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

Most results for Al, Ca and Mg were obtained by atomic absorption on a single solution prepared from a separate subsample of CCU-1. Approximately one half of the contributors 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 a few sets of results for each of Al_2O_3 and CaO 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 the gangue minerals.

Silica

All but one of the 15 contributing laboratories determined silica gravimetrically by common procedures. Approximately one half used a direct sodium peroxide-hydroxide fusion for sample decomposition. The other half used a mixed-acid treatment with the residue being fused with sodium carbonate in most cases. Silica was dehydrated with perchloric or sulphuric acid and either weighed directly or determined by difference after volatilization with hydrofluoric acid.

Lead

All but three laboratories used atomic absorption for determining lead. Most of these used a mixed-acid decomposition involving hydrofluoric acid. The final solution was either dilute hydrochloric, perchloric or nitric acid and in most cases it was analyzed for zinc as well (Table 5).

Mercury

The cold-vapour atomic absorption method was used by 12 of 13 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 all participants analyzed subsamples that had been dried previously at 105°C for 2 h.

Stability of CCU-1

Figure 2 shows the laboratory means for copper in CCU-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 CCU-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 CCU-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 CCU-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 CCU-1.

Figure 3 also shows that unprotected test bottles of CCU-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 CCU-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

<u>Recommended values and associated statistical parameters</u>
(outliers excluded)

Element (oxide)	N	n	$\bar{x}$	95% CL low	95% CL high	Spread %	$\overline{cv}$ %	CF
			(wt %)	(wt %)	(wt %)			
Al_2O_3	15	160	0.247	0.240	0.253	5.2	3.1	1.7
Cu	35	368	24.71	24.67	24.76	0.4	0.2	1.6
Pb	29	298	0.106	0.102	0.111	8.9	3.1	2.8
SiO_2	15	133	2.61	2.53	2.68	5.7	1.8	3.1
Zn	25	258	3.22	3.19	3.26	2.3	0.9	2.6
			(µg/g)	(µg/g)	(µg/g)			
Ag	27	248	139	136	142	4.5	1.4	3.2
Au	20	185	7.5	7.2	7.8	6.8	3.1	2.2
Hg	13	130	61	59	63	7.7	2.8	2.7

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	140	0.09	0.08	0.10	17	4	4
Fe	5	34	30.8	30.6	31.0	1.3	0.2	7
MgO	14	149	1.11	1.06	1.16	9	1.6	6
S	4	30	35.6	35.3	35.8	1.4	0.6	3*
			(µg/g)	(µg/g)	(µg/g)			
As	10	101	42	35	49	32	8	4
Bi	3	23	26	8	44	136	14	10
Cd	2	25	109	--	--	--	--	--
Mo	2	15	35	--	--	--	--	--
Se	8	78	121	111	132	18	4	4
Te	7	68	19	12	27	79	10	8

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 less than 4; however, S not certified because of insufficient data.

TABLE 5(a)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (μg/g)
Atomic absorption	HNO_3 + Br_2 + HCl (one with HF); final solution 25-50% v/v HCl	5	6,21b,27,35, 38	50	139
	HNO_3 + HCl; final solution 25% v/v HCl	2	21a,37	16	140
	HNO_3 + HF + $HClO_4$; final solution 3-5% v/v $HClO_4$	1	26	10	139
	$HClO_4$ + HF; final solution 10% v/v $HClO_4$	1	14	10	138
	HNO_3 + Br_2 + H_2SO_4; final solution dilute tartaric + HNO_3 acids	1	20	10	137
	HNO_3 + HF; final solution 1N HNO_3	1	29	10	140
	HNO_3; Ag ext'd into MIBK with iso-octylthiophosphate	1	9	10	134
	HNO_3 + Br_2; other details not given	1	41	4	144
	Details not given	1	30	10	152
Fire assay	Classical fire assay; gravimetric finish	7	5,10,12,19, 25,30,43	64	138
	HNO_3 decomposition; Ag + Pb ppt'd, assayed to give bead	2	40,41	14	140
Fire assay-atomic absorption	Pb button scorified to ∿ 2 g, dissolved in HNO_3 for a.a.	1	2	10	142
	Pb button partially cupelled, dissolved in HNO_3 for a.a.	1	17	10	141
	Sample digested in HNO_3, soln analyzed by a.a.; residue assayed to give Pb button which was dissolved in HNO_3 for a.a.	1	17	10	140
	Pb button cupelled to ∿ 100 mg; bead fused with Na_2O_2 + CuO; cake dissolved and soln treated with Cd-Cu releasing reagent for a.a.	1	21	10	131

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

TABLE 5(b)

Summary of methods for Au in CCU-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (μg/g)
Fire assay					
	Classical fire assay	7	5,10,12,14,19, 25,43	70	7.6
	Classical fire assay of residue after HNO_3 treatment of sub-sample	2	27,40	12	6.9
Fire assay-atomic abs.					
	Classical fire assay; Pb button scorified to ∿ 2 g, treated with HNO_3; residue dissolved in aqua regia for a.a. detn	1	2	10	6.8
	Classical fire assay; 100 mg Pb button fused with Na_2O_2 + CuO	1	21	10	7.5
	Classical fire assay; Ag bead dissolved in aqua regia for a.a.	1	29	10	7.4
Fire assay-emm.spec.					
	Classical fire assay; Au collected in Ag bead; analyzed by emission spectroscopy	2	17,44	19	7.2
Atomic absorption					
	Sample digested in aqua regia (one also with Br_2); Au ext'd with MIBK for a.a.	4	6,9,21a,35	34	8.1
	Sample digested in aqua regia; Au collected with Te°; dissolved in aqua regia for a.a.	1	20	10	7.7
	Sample digested in aqua regia; Au ext'd as bromide with diethyl ether	1	2	10	7.1

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

TABLE 5(c)

Summary of analytical methods for Cu in CCU-1 (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Electrolytic					
	$HClO_4$; Cu electrodeposited from dil. HNO_3-H_2SO_4 "plating acid"	2	21a,21b	14	24.71
	HNO_3 + $KClO_4$ + Br_2 + H_2SO_4; as above	2	6,32	20	24.75
	HNO_3 + HF + H_2SO_4; as above	2	10,17	20	24.84
	HNO_3 + Br_2 + H_2SO_4; as above (soln contained sulphamic acid)	1	30	11	24.66
	HNO_3 + HCl + Br_2 + H_2SO_4; as above (soln contained urea, tartaric and/or hydrazine)	2	35,38	20	24.75
	HNO_3 + HCl + Br_2 + H_2SO_4; Cu plated from soln containing hydrazine, amm. sulphate and urea	1	12	10	24.71
	Br_2 + H_2SO_4; Fe removed with NH_3; filtrate electrolyzed	1	27	10	24.94
	HNO_3 + Br_2 + HF + H_2SO_4; Cu^+ sep'd as thiocyanate, dissolved in H_2SO_4 and HNO_3 and electrolyzed	2	19,23	22	24.63
	HNO_3 + HCl + H_2SO_4; As, Sb and Pb removed before electrolysis	1	21c	4	24.57
	Oxidizing acid mixture; other details not given	2	21,41	20	24.63
	Details not given	3	4,25,50	25	24.85
Titrimetric					
Short iodide method	Various acid mixtures involving one or more of HNO_3, Br_2, HF, HCl, H_2SO_4, $HClO_4$, $KClO_4$ (see Appendix B)	8	1,5,12,20,30, 43,44,46	102	24.65
Long iodide method	HNO_3 + HCl + H_2SO_4 (see Appendix B)	5	4,14,36,37,40	50	24.70
Atomic absorption					
	HNO_3 + HF + $HClO_4$; final soln 10% HCl	1	7	20	24.85
	HNO_3 + Br_2 + HF + H_2SO_4; dilution with H_2O	1	20	10	24.53
	HNO_3 + HCl + HF + $HClO_4$; final soln 5% $HClO_4$, slanted burner	1	26	10	24.91

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

TABLE 5(d)

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

Method	Decomposition, separations, etc.	N	Lab no.	n	$\bar{x}$ (wt %)
Atomic absorption					
	HNO_3 + one or more of Br_2, HCl, HF, $HClO_4$; final solution 2-25% v/v HCl	7	5,6,7,21a, 21b,35,40	76	3.27
	HNO_3 + HCl + one of Br_2, HF, $HClO_4$; final solution 3-6% v/v HNO_3	3	10,29,38	30	3.24
	Na_2O_2 fusion; final solution 3% v/v HNO_3	1	27	10	3.33
	$HClO_4$ + HF; final solution 5% v/v $HClO_4$	1	14	10	3.24
	HNO_3 + HCl + HF + $HClO_4$; final solution 3-5% v/v $HClO_4$	1	26	10	3.21
	Various oxidizing acid mixtures; other details not given	4	9,12,20,44	40	3.16
	HNO_3 + HCl + $HClO_4$ or H_2SO_4; Cu removed by electrolysis, Zn det'd in electrolyte	2	21,41	22	3.14
	Acid decomp; no other details	1	25	10	3.09
Titrimetric					
EDTA	HNO_3 + HBr + $HClO_4$ + H_2SO_4; Zn sep'd from matrix elements by anion exchange	1	1	10	3.29
	HNO_3 + HCl + $HClO_4$ + H_2SO_4; Zn sep'd from matrix elements by extn with MIBK	1	24	10	3.21
Ferrocyanide	HNO_3 + HCl + H_2SO_4; Zn sep'd as sulphide	1	36	10	3.13
	No details given	1	19	10	3.34
Ferrocyanide-amperometric	Br_2 in CCl_4 + HNO_3 + HCl + H_2SO_4; Fe ppt'd with NH_3, Cu removed with Pb°	1	1	10	3.21

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	140	0.092	17	4
Fe	TITR	5	34	30.8	1.3	0.2
MgO	AA	14	149	1.11	9	1.6
S	GRAV	4	30	35.6	1.4	0.6
				(µg/g)		
As	AA	1	8	25	--	12
	COLOR	8	81	43	34	8
	NAA	1	12	45	--	5
Bi	AA	1	8	22	--	20
	COLOR	1	10	32	--	4
	ES	1	5	20	--	18
Cd	AA	1	20	116	--	4
	ES	1	5	79	--	20
Mo	AA	1	10	40	--	33
	ES	1	5	24	--	17
Se	AA	3	28	127	57	4
	COLOR	4	40	115	33	5
	XRF	1	10	128	--	3
Te	AA	3	28	19	350	12
	COLOR	2	20	18	--	5
	XRF	2	20	21	--	13

AA = atomic absorption; TITR = titrimetric; COLOR = colorimetric (spectrophotometric); GRAV = gravimetric; ES = emission spectrographic; NAA = neutron activation analysis; XRF = X-ray fluorescence.

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- 2	(FA-AA)	5	141.8000	1.3038	5	142.4000	1.5166	A	10	142.1000	1.3703	.96
LAB- 5	(FA)	5	122.0000	1.5811	5	119.6000	1.1402	REJECT	10	120.8000	1.8135	1.50
LAB- 6	(AA)	5	139.8000	1.0954	5	138.0000	1.7321	A	10	138.9000	1.6633	1.20
LAB- 9	(AA)	5	135.8000	1.0954	5	133.1000	2.2192	REJECT	10	134.4500	2.1788	1.62
LAB-10	(FA)	5	146.6000	1.5166	5	144.0000	2.0000	REJECT	10	145.3000	2.1628	1.49
LAB-12	(FA)	5	144.4000	1.3416	5	144.4000	.8944	A	10	144.4000	1.0750	.74
LAB-14	(AA)	5	137.9000	1.2268	5	139.0800	.6221	A	10	138.4900	1.1080	.80
LAB-17	(AA)	5	139.8000	1.0954	5	140.4000	2.1909	A	10	140.1000	1.6633	1.19
LAB-17	(FA-AA)	5	140.8000	4.1473	5	141.8000	2.1679	A	10	141.3000	3.1640	2.24
LAB-19	(FA)	5	140.8200	3.7352	5	139.1800	2.0669	A	10	140.0000	2.9743	2.12
LAB-20	(AA)	5	137.0741	1.3065	5	136.8737	.8963	A	10	136.9739	1.0615	.77
LAB-21	(AA)	5	124.2485	.7085	5	126.2525	1.0020	REJECT	10	125.2505	1.3360	1.07
LAB-21	(AA)	3	155.6446	2.3140	3	151.6366	.5785	REJECT	6	153.6406	2.6636	1.73
LAB-21	(FA-AA)	5	130.2000	2.2528	5	131.1000	.8944	A	10	130.6500	1.6841	1.29
LAB-25	(FA)	5	126.4600	1.2361	5	124.8000	.7000	REJECT	10	125.6300	1.2893	1.03
LAB-26	(AA)	5	138.0000	4.4721	5	140.0000	0.0000	A	10	139.0000	3.1623	2.28
LAB-27	(AA)	5	142.6000	.5477	5	142.0000	0.0000	***R**	10	142.3000	.4830	.34
LAB-29	(AA)	5	139.6000	1.1402	5	139.8000	2.2804	A	10	139.7000	1.7029	1.22
LAB-30	(FA)	2	145.6413	3.8969	2	140.8317	2.9049	A	4	143.2365	3.9478	2.76
LAB-30	(AA)	5	151.8437	1.1013	5	152.6653	.5424	A	10	152.2545	.9259	.61
LAB-35	(AA)	5	146.3527	3.0864	5	145.6513	1.8660	A	10	146.0020	2.4327	1.67
LAB-37	(AA)	5	131.2000	.8367	5	131.0000	0.0000	A	10	131.1000	.5676	.43
LAB-38	(AA)	5	144.0000	5.4772	5	142.0000	4.4721	A	10	143.0000	4.8305	3.38
LAB-40	(FA)	5	139.1800	3.3885	5	140.3000	2.6907	A	10	139.7400	2.9444	2.11
LAB-41	(FA)	2	141.8000	2.6870	2	142.0000	.4243	A	4	141.9000	1.5748	1.11
LAB-41	(AA)	2	144.5000	.7071	2	144.0000	1.4142	A	4	144.2500	.9574	.66
LAB-43	(FA)	5	146.8000	1.6432	5	146.8000	1.6432	A	10	146.8000	1.5492	1.06
LAB-46	(ES)	THERE IS ONLY 1 BOTTLE							5	87.0000	10.3682	11.92
LAB-50	(AA)	5	360.0000	0.0000	5	340.0000	14.1421	***R**	10	350.0000	14.1421	4.04

Variance between sets, between bottles and within bottles = 5.62×10^{1}, 1.46×10^{-1} and 4.41, 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- 5	(AA)	5	.2555	.0134	5	.2487	.0089	A	10	.2521	.0113	4.48
LAB- 6	(GRAV)	5	.2358	.0158	5	.2143	.0175	A	10	.2250	.0194	8.62
LAB-10	(AA)	5	.2683	.0085	5	.2456	.0000	***R**	10	.2569	.0132	5.14
LAB-12	(AA)	5	.2441	.0021	5	.2456	.0013	A	10	.2449	.0018	.74
LAB-14	(AA)	5	.2645	.0000	5	.2645	.0000	A	10	.2645	.0000	.00
LAB-17	(AA)	5	.2392	.0017	5	.2385	.0021	A	10	.2389	.0018	.77
LAB-19	(AA)	5	.2475	.0042	5	.2419	.0042	A	10	.2447	.0050	2.02
LAB-23	(AA)	10	.2479	.0092	10	.2568	.0169	A	20	.2523	.0140	5.57
LAB-24	(AA)	5	.2517	.0061	5	.2494	.0045	A	10	.2505	.0052	2.06
LAB-25	(AA)	5	.2456	.0000	5	.2456	.0000	A	10	.2456	.0000	.00
LAB-26	(AA)	5	.2362	.0060	5	.2339	.0053	A	10	.2351	.0055	2.32
LAB-35	(AA)	5	.2612	.0051	5	.2556	.0000	***R**	10	.2584	.0045	1.76
LAB-40	(AA)	5	.2555	.0016	5	.2604	.0036	REJECT	10	.2579	.0037	1.43
LAB-41	(AA)	2	.2022	.0107	2	.2126	.0040	A	4	.2074	.0089	4.30
LAB-44	(AA)	5	.2313	.0090	5	.2233	.0043	A	10	.2273	.0079	3.47
LAB-50	(AA)	5	.2380	.0104	5	.2418	.0246	A	10	.2399	.0179	7.47

Variance between sets, between bottles and within bottles = 1.16×10^{-4}, 3.79×10^{-5}, and 7.54×10^{-5}, respectively.

TABLE 7 (cont'd)

Au (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	(FA-AA)	5	6.6260	.1743	5	6.9480	.1108	REJECT	10	6.7870	.2185	3.22
LAB- 2	(AA)	5	7.2220	.1871	5	7.0460	.2029	A	10	7.1340	.2061	2.89
LAB- 5	(FA)	5	8.3200	.2280	5	8.1200	.1789	A	10	8.2200	.2201	2.68
LAB- 6	(AA)	5	7.1400	.2563	5	7.0300	.1848	A	10	7.0850	.2185	3.08
LAB- 9	(AA)	5	8.6200	.0975	5	8.7100	.1517	A	10	8.6650	.1292	1.49
LAB-10	(FA)	5	7.7200	.4087	5	7.7400	.2074	A	10	7.7300	.3057	3.95
LAB-12	(FA)	5	7.7080	.1272	5	7.5960	.0586	A	10	7.6520	.1104	1.44
LAB-14	(FA)	5	6.9260	.3752	5	8.0920	.3086	REJECT	10	7.5090	.6947	9.25
LAB-17	(FA-ES)	5	7.2320	.2540	5	7.2120	.1942	A	10	7.2220	.2134	2.96
LAB-19	(FA)	5	7.4060	.1060	5	7.4060	.2166	A	10	7.4060	.1608	2.17
LAB-20	(AA)	5	7.7555	.2413	5	7.7154	.2744	A	10	7.7354	.2445	3.16
LAB-21	(AA)	2	8.1413	.0921	2	8.3818	.1913	A	4	8.2615	.1852	2.24
LAB-21	(FA-AA)	5	7.5200	.0837	5	7.4000	.1871	A	10	7.4600	.1506	2.02
LAB-25	(FA)	5	7.5060	.0760	5	7.5060	.0760	A	10	7.5060	.0717	.95
LAB-27	(FA)	5	6.8360	.1045	5	6.8100	.0875	A	10	6.8230	.0919	1.35
LAB-29	(FA-AA)	5	7.2600	.2074	5	7.5200	.1789	A	10	7.3900	.2283	3.09
LAB-35	(AA)	5	8.4088	.1222	5	8.4489	.0818	A	10	8.4288	.1003	1.19
LAB-40	(FA)	INSUFFICIENT DATA							2	7.3050	.1061	1.45
LAB-41	(FA)	INSUFFICIENT DATA							2	6.0850	.2051	3.37
LAB-43	(FA)	5	6.8400	.1517	5	6.8600	.1342	A	10	6.8500	.1354	1.98
LAB-44	(FA-ES)	4	6.9250	.6500	5	7.4000	.8337	A	9	7.1889	.7541	10.49

Variance between sets, between bottles and within bottles = 2.67×10^{-1}, 4.10×10^{-2} and 3.84×10^{-2}, 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	(TITR)	THERE ARE MORE THAN 2 BOTTLES							30	24.7303	.0652	.26
LAB- 4	(TITR)	5	24.7540	.0288	5	24.7280	.0415	A	10	24.7410	.0363	.15
LAB- 4	(ELECTR)	THERE IS ONLY 1 BOTTLE							5	24.7460	.0336	.14
LAB- 5	(TITR)	5	24.5820	.0335	5	24.5580	.0249	A	10	24.5700	.0306	.12
LAB- 6	(ELECTR)	5	24.6920	.0164	5	24.6900	.0141	A	10	24.6910	.0145	.06
LAB- 7	(AA)	THERE ARE MORE THAN 2 BOTTLES							20	24.8470	.2655	1.07
LAB- 9	(TITR)	5	23.4520	.0327	5	23.7200	.0308	REJECT	10	23.5860	.1444	.61
LAB-10	(ELECTR)	5	24.9040	.0152	5	24.8660	.0152	REJECT	10	24.8850	.0246	.10
LAB-12	(TITR)	5	24.7500	.0975	5	24.6960	.0472	A	10	24.7230	.0776	.31
LAB-12	(ELECTR)	5	24.7140	.0055	5	24.7020	.0130	A	10	24.7080	.0114	.05
LAB-14	(TITR)	5	24.7300	.0071	5	24.7400	.0100	A	10	24.7350	.0097	.04
LAB-17	(ELECTR)	5	24.8240	.0329	5	24.7680	.0228	REJECT	10	24.7960	.0398	.16
LAB-19	(ELECTR)	5	24.6060	.0207	5	24.5940	.0251	A	10	24.6000	.0226	.09
LAB-20	(AA)	5	24.5511	.1863	5	24.5030	.2576	A	10	24.5271	.2134	.87
LAB-20	(TITR)	5	24.4048	.1334	5	24.6192	.0888	REJECT	10	24.5120	.1555	.63
LAB-21	(ELECTR)	2	24.5842	.0213	2	24.5641	.0071	A	4	24.5741	.0174	.07
LAB-21	(ELECTR)	5	24.7315	.0179	5	24.6894	.0246	REJECT	10	24.7104	.0301	.12
LAB-21	(ELECTR)	2	24.7746	.0213	2	24.6293	.0142	REJECT	4	24.7019	.0852	.34
LAB-21	(ELECTR)	5	24.6280	.0130	5	24.6260	.0336	A	10	24.6270	.0241	.10
LAB-23	(ELECTR)	5	24.6620	.0045	5	24.6640	.0055	A	10	24.6630	.0048	.02
LAB-25	(ELECTR)	5	24.7460	.0261	5	24.7200	.0212	A	10	24.7330	.0263	.11
LAB-26	(AA)	5	24.9320	.0593	5	24.8960	.0550	A	10	24.9140	.0572	.23
LAB-27	(ELECTR)	5	24.9300	.0212	5	24.9500	.0283	A	10	24.9400	.0258	.10
LAB-29	(AA)	4	24.4500	.1000	5	24.3200	.0837	A	9	24.3778	.1093	.45
LAB-30	(ELECTR)	6	24.6660	.0558	5	24.6473	.0148	A	11	24.6575	.0417	.17
LAB-30	(TITR)	8	24.6869	.0918	9	24.6994	.0792	A	17	24.6935	.0829	.34
LAB-32	(ELECTR)	5	24.8320	.0554	5	24.7940	.0477	A	10	24.8130	.0527	.21
LAB-35	(ELECTR)	5	24.7655	.0251	5	24.8156	.0168	REJECT	10	24.7906	.0332	.13
LAB-36	(TITR)	5	24.6280	.0370	5	24.6000	.0308	A	10	24.6140	.0353	.14
LAB-37	(TITR)	5	24.7900	.0224	5	24.7900	.0224	A	10	24.7900	.0211	.09
LAB-38	(ELECTR)	5	24.6680	.0432	5	24.7440	.0635	A	10	24.7060	.0650	.26
LAB-39	(AA)	5	25.2900	.0758	5	25.3020	.0409	A	10	25.2960	.0578	.23
LAB-40	(TITR)	5	24.6420	.0192	5	24.5620	.0311	REJECT	10	24.6020	.0487	.20
LAB-41	(ELECTR)	6	24.6283	.0741	6	24.6300	.0126	A	12	24.6292	.0507	.21
LAB-43	(TITR)	5	24.6080	.0217	5	24.5940	.0261	A	10	24.6010	.0238	.10
LAB-44	(TITR)	5	24.6240	.0737	5	24.5620	.0567	A	10	24.5930	.0701	.28
LAB-46	(TITR)	THERE IS ONLY 1 BOTTLE							5	24.4400	.0600	.25
LAB-46	(TITR)	THERE IS ONLY 1 BOTTLE							5	24.2540	.2315	.95
LAB-50	(ELECTR)	5	24.9860	.0537	5	25.0280	.0589	A	10	25.0070	.0576	.23

Variance between sets, between bottles and within bottles = 1.31×10^{-2}, 4.33×10^{-4} and 6.00×10^{-3}, respectively.

TABLE 7 (cont'd)
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- 4 (AA)	5	65.6000	1.9494	5	62.0000	1.4142	REJECT	10	63.8000	2.4855	3.90
LAB- 5 (AA)	5	60.0200	1.0592	5	59.7000	1.0724	A	10	59.8600	1.0189	1.70
LAB- 6 (AA)	5	60.8000	1.7664	5	62.7200	.8672	A	10	61.7600	1.6568	2.68
LAB-12 (AA)	5	64.2400	1.7981	5	63.9800	.8701	A	10	64.1100	1.3387	2.09
LAB-14 (AA)	5	65.8000	2.6833	5	66.4000	1.6733	A	10	66.1000	2.1318	3.23
LAB-19 (AA)	5	54.6000	.5477	5	53.8000	.8367	A	10	54.2000	.7888	1.46
LAB-20 (AA)	5	61.9238	3.8418	5	67.1343	2.8341	REJECT	10	64.5291	4.2037	6.51
LAB-23 (AA)	5	83.4000	2.5100	5	79.2000	2.2804	REJECT	10	81.3000	3.1640	3.89
LAB-25 (AA)	5	63.2000	.8367	5	61.8000	1.3038	A	10	62.5000	1.2693	2.03
LAB-26 (AA)	5	64.9000	1.0223	5	64.8800	1.7021	A	10	64.8900	1.3237	2.04
LAB-27 (AA)	5	93.2000	2.6833	5	84.8000	7.9498	A	10	89.0000	7.1336	8.02
LAB-29 (AA)	5	58.2600	.7893	5	59.4200	.8556	A	10	58.8400	.9879	1.68
LAB-37 (AA)	5	59.9600	.8444	5	58.9400	.6804	A	10	59.4500	.9009	1.52
LAB-40 (COLOR)	5	60.6000	1.5572	5	58.6800	1.3027	A	10	59.6400	1.6900	2.83
LAB-44 (AA)	5	52.9200	2.1902	5	54.7800	2.8735	A	10	53.8500	2.6005	4.83
LAB-46 (ES)	THERE IS ONLY 1 BOTTLE							5	82.0000	19.2354	23.46

Variance between sets, between bottles and within bottles = 1.40 x 10^1, 1.59 and 2.91 respectively.

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	.1080	.0045	5	.1060	.0055	A	10	.1070	.0048	4.51
LAB- 5 (AA)	5	.1072	.0058	5	.1016	.0066	A	10	.1044	.0066	6.28
LAB- 6 (AA)	5	.1000	.0000	5	.1000	.0000	A	10	.1000	.0000	.00
LAB- 7 (AA)	THERE ARE MORE THAN 2 BOTTLES							20	.1283	.0056	4.40
LAB- 9 (AA)	5	.0904	.0005	5	.0901	.0011	A	10	.0902	.0008	.91
LAB-10 (AA)	5	.1100	.0000	5	.1000	.0000	***R**	10	.1050	.0053	5.02
LAB-12 (AA)	5	.1016	.0009	5	.1008	.0004	A	10	.1012	.0008	.78
LAB-14 (AA)	5	.1300	.0000	5	.1300	.0000	A	10	.1300	.0000	.00
LAB-19 (AA)	5	.1036	.0013	5	.1050	.0012	A	10	.1043	.0014	1.36
LAB-20 (AA)	5	.1086	.0015	5	.1128	.0018	REJECT	10	.1107	.0027	2.46
LAB-21 (AA)	5	.0960	.0008	5	.0980	.0023	A	10	.0970	.0019	1.99
LAB-21 (AA)	3	.1310	.0023	3	.1303	.0020	A	6	.1306	.0020	1.51
LAB-21 (AA)	5	.1040	.0089	5	.0940	.0055	A	10	.0990	.0088	8.84
LAB-23 (AA)	10	.1180	.0169	10	.1250	.0151	A	20	.1215	.0160	13.16
LAB-25 (AA)	5	.1000	.0000	5	.1000	.0000	A	10	.1000	.0000	.00
LAB-26 (AA)	5	.1084	.0009	5	.1082	.0008	A	10	.1083	.0008	.76
LAB-27 (AA)	5	.1102	.0011	5	.1120	.0000	***R**	10	.1111	.0012	1.08
LAB-29 (AA)	5	.1036	.0036	5	.1026	.0038	A	10	.1031	.0035	3.44
LAB-35 (AA)	5	.0946	.0017	5	.0946	.0017	A	10	.0946	.0016	1.67
LAB-36 (GRAV)	5	.0860	.0089	5	.0900	.0071	A	10	.0880	.0079	8.96
LAB-37 (AA)	5	.1006	.0013	5	.1030	.0012	REJECT	10	.1018	.0018	1.72
LAB-38 (AA)	5	.0990	.0022	5	.1000	.0000	A	10	.0995	.0016	1.59
LAB-39 (AA)	5	.1160	.0032	5	.1144	.0005	A	10	.1152	.0023	2.00
LAB-40 (AA)	5	.1072	.0008	5	.1066	.0005	A	10	.1069	.0007	.69
LAB-41 (AA)	6	.0902	.0021	6	.0892	.0013	A	12	.0897	.0018	1.98
LAB-41 (AA)	3	.0917	.0021	3	.0893	.0012	A	6	.0905	.0020	2.18
LAB-44 (AA)	5	.1058	.0018	5	.1046	.0018	A	10	.1052	.0018	1.72
LAB-46 (GRAV)	THERE IS ONLY 1 BOTTLE							4	.0925	.0096	10.35
LAB-46 (GRAV)	THERE IS ONLY 1 BOTTLE							5	.2020	.0084	4.14
LAB-50 (AA)	5	.1080	.0024	5	.1068	.0011	A	10	.1074	.0019	1.77

Variance between sets, between bottles and within bottles = 1.16 x 10^{-4}, 5.33 x 10^{-6}, and 1.55 x 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- 6	(GRAV)	5	2.6955	.0262	5	2.7640	.0464	REJECT	10	2.7297	.0506	1.86
LAB-10	(GRAV)	5	1.0141	.0325	5	.9627	.0000	***R**	10	.9884	.0347	3.51
LAB-12	(GRAV)	5	2.5972	.0287	5	2.5758	.0325	A	10	2.5865	.0310	1.20
LAB-14	(GRAV)	5	2.5544	.0469	5	2.5030	.0000	***R**	10	2.5287	.0413	1.64
LAB-19	(GRAV)	5	2.6271	.0096	5	2.6485	.0317	A	10	2.6378	.0248	.94
LAB-23	(GRAV)	5	2.5201	.0464	5	2.5244	.0400	A	10	2.5223	.0409	1.62
LAB-24	(GRAV)	5	2.6014	.0259	5	2.6806	.1111	A	10	2.6410	.0868	3.28
LAB-25	(GRAV)	5	2.7854	.0412	5	2.7597	.0151	A	10	2.7725	.0322	1.16
LAB-26	(AA)	5	2.6357	.0234	5	2.6442	.0191	A	10	2.6400	.0207	.78
LAB-27	(GRAV)	4	2.5886	.0175	5	2.5629	.0613	A	9	2.5743	.0466	1.81
LAB-35	(GRAV)	5	2.3837	.0701	5	2.5295	.0215	REJECT	10	2.4566	.0911	3.71
LAB-36	(GRAV)	5	2.5800	.0358	5	2.5629	.0488	A	10	2.5715	.0413	1.61
LAB-40	(GRAV)	5	2.4174	.0502	5	2.3575	.0666	A	10	2.3875	.0639	2.68
LAB-41	(GRAV)	2	2.6207	.0151	2	2.5993	.0151	A	4	2.6100	.0175	.67
LAB-46	(GRAV)	THERE IS ONLY 1 BOTTLE							5	2.9779	.0593	1.99
LAB-46	(GRAV)	THERE IS ONLY 1 BOTTLE							5	2.7597	.0741	2.68
LAB-50	(GRAV)	5	3.2860	.2216	5	3.3716	.0940	A	10	3.3288	.1667	5.01

Variance between sets, between bottles and within bottles = 1.19×10^{-2}, 1.44×10^{-3} and 1.98×10^{-3}, 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	(TITR)	5	3.2880	.0084	5	3.2920	.0045	A	10	3.2900	.0067	.20
LAB- 1	(TITR)	5	3.2040	.0182	5	3.2100	.0235	A	10	3.2070	.0200	.62
LAB- 5	(AA)	5	3.1296	.0146	5	3.1096	.0174	A	10	3.1196	.0184	.59
LAB- 6	(AA)	5	3.3180	.0084	5	3.3360	.0152	REJECT	10	3.3270	.0149	.45
LAB- 7	(AA)	THERE ARE MORE THAN 2 BOTTLES							20	3.2947	.0333	1.01
LAB- 9	(AA)	5	3.1700	.0274	5	3.1700	.0308	A	10	3.1700	.0275	.87
LAB-10	(AA)	5	3.2420	.0045	5	3.2420	.0148	A	10	3.2420	.0103	.32
LAB-12	(AA)	5	3.1480	.0342	5	3.1680	.0179	A	10	3.1580	.0278	.88
LAB-14	(AA)	5	3.2400	.0548	5	3.2400	.0548	A	10	3.2400	.0516	1.59
LAB-19	(TITR)	5	3.3320	.0110	5	3.3380	.0205	A	10	3.3350	.0158	.47
LAB-20	(AA)	5	3.0922	.0208	5	3.0682	.0110	A	10	3.0802	.0202	.65
LAB-21	(AA)	5	3.3126	.0090	5	3.3287	.0131	A	10	3.3207	.0135	.41
LAB-21	(AA)	3	3.4202	.0232	3	3.4402	.0417	A	6	3.4302	.0321	.94
LAB-21	(AA)	5	3.2180	.0192	5	3.2220	.0164	A	10	3.2200	.0170	.53
LAB-23	(AA)	10	3.3800	.1033	10	3.4100	.1101	A	20	3.3950	.1050	3.09
LAB-24	(TITR)	5	3.2240	.0288	5	3.2040	.0559	A	10	3.2140	.0433	1.35
LAB-25	(AA)	5	3.0880	.0438	5	3.0880	.0438	A	10	3.0880	.0413	1.34
LAB-26	(AA)	5	3.2120	.0192	5	3.1980	.0130	A	10	3.2050	.0172	.54
LAB-27	(TITR)	5	3.5240	.0182	5	3.5300	.0316	A	10	3.5270	.0245	.70
LAB-27	(AA)	5	3.3200	.0212	5	3.3400	.0200	A	10	3.3300	.0221	.66
LAB-29	(AA)	5	3.2800	.0324	5	3.2760	.0385	A	10	3.2780	.0336	1.02
LAB-35	(AA)	5	3.3147	.0269	5	3.2184	.0628	REJECT	10	3.2666	.0681	2.09
LAB-36	(TITR)	5	3.1340	.0251	5	3.1240	.0167	A	10	3.1290	.0208	.66
LAB-38	(AA)	5	3.1960	.0207	5	3.1780	.0192	A	10	3.1870	.0211	.66
LAB-39	(AA)	5	4.2220	.0311	4	4.2275	.0206	A	9	4.2244	.0255	.60
LAB-40	(AA)	5	3.1980	.0148	5	3.2080	.0239	A	10	3.2030	.0195	.61
LAB-41	(AA)	6	3.0767	.0979	6	3.0517	.0637	A	12	3.0642	.0798	2.61
LAB-44	(AA)	5	3.2360	.0428	5	3.2240	.0251	A	10	3.2300	.0337	1.04
LAB-46	(TITR)	THERE IS ONLY 1 BOTTLE							5	2.7220	.1238	4.55
LAB-46	(TITR)	THERE IS ONLY 1 BOTTLE							5	3.0340	.0532	1.75

Variance between sets, between bottles and within bottles = 6.52×10^{-3}, 8.78×10^{-5} and 1.09×10^{-3}, respectively.

TABLE 8

Analytical results for reference concentrate CCU-1

Ag (μg/g)

LAB- 6 (AA)	139	141	141	139	139	141	137	137	137	138
LAB- 9 (AA)	137	135	137	135	135	135	135	130	135	132
LAB-14 (AA)	137	138	138	137	140	139	139	140	139	139
LAB-17 (AA)	141	140	140	140	138	139	139	144	139	141
LAB-20 (AA)	138	137	138	135	136	136	138	136	136	137
LAB-21 (AA)	124	124	125	124	123	127	125	127	125	126
LAB-21 (AA)	158	154	154	152	151	151				
LAB-26 (AA)	130	140	140	140	140	140	140	140	140	140
LAB-27 (AA)	143	143	142	143	142	142	142	142	142	142
LAB-29 (AA)	139	138	140	141	140	137	138	140	142	142
LAB-30 (AA)	152	153	150	152	153	153	153	153	152	153
LAB-35 (AA)	144	144	148	151	144	144	148	148	144	144
LAB-37 (AA)	132	130	131	131	132	131	131	131	131	131
LAB-38 (AA)	140	150	150	140	140	140	140	150	140	140
LAB-41 (AA)	145	144	143	145						
* LAB-46 (ES)	75	85	95	80	100					
LAB- 5 (FA)	123	124	120	122	121	118	121	119	120	120
LAB-10 (FA)	147	149	146	145	146	147	143	143	142	145
LAB-12 (FA)	146	143	143	145	145	144	145	145	145	143
LAB-19 (FA)	137	142	139	147	140	142	137	137	140	140
LAB-25 (FA)	127	128	126	126	125	124	126	124	125	126
LAB-30 (FA)	143	148	143	139						
LAB-40 (FA)	142	140	136	135	142	139	143	137	141	143
LAB-41 (FA)	140	144	142	142						
LAB-43 (FA)	145	145	148	148	148	145	145	148	148	148
LAB- 2 (FA-AA)	144	141	141	142	141	141	144	144	142	141
LAB-17 (FA-AA)	138	136	140	146	144	139	140	143	144	143
LAB-21 (FA-AA)	134	130	130	129	129	131	131	133	132	131

Al_2O_3 (wt %)

LAB- 5 (AA)	.255	.236	.257	.255	.274	.246	.240	.261	.255	.242
LAB-10 (AA)	.283	.265	.265	.265	.265	.246	.246	.246	.246	.246
LAB-12 (AA)	.244	.248	.242	.244	.244	.244	.248	.246	.246	.246
LAB-14 (AA)	.265	.265	.265	.265	.265	.265	.265	.265	.265	.265
LAB-17 (AA)	.240	.236	.240	.240	.240	.240	.236	.236	.240	.240
LAB-19 (AA)	.249	.240	.249	.249	.249	.249	.240	.240	.240	.240
LAB-23 (AA)	.231	.249	.249	.261	.261	.240	.249	.249	.240	.249
	.249	.280	.280	.280	.261	.240	.249	.249	.240	.240
LAB-24 (AA)	.259	.255	.248	.244	.253	.253	.246	.248	.246	.255
LAB-25 (AA)	.246	.246	.246	.246	.246	.246	.246	.246	.246	.246
LAB-26 (AA)	.227	.234	.238	.240	.242	.236	.231	.242	.229	.232
LAB-35 (AA)	.256	.256	.265	.265	.265	.256	.256	.256	.256	.256
LAB-40 (AA)	.255	.257	.253	.257	.255	.259	.266	.257	.259	.261
* LAB-41 (AA)	.195	.210	.210	.215						
LAB-44 (AA)	.231	.238	.219	.227	.242	.229	.225	.225	.221	.217
LAB-50 (AA)	.227	.227	.246	.246	.246	.265	.208	.246	.227	.265
LAB- 6 (GRAV)	.242	.251	.234	.242	.210	.217	.193	.200	.234	.227

As (μg/g)

LAB-50 (AA)	24	22	29	23	30	29	23	23		
LAB- 3 (COLOR)	44	44	42	40	44	46	45	46	46	47
LAB- 6 (COLOR)	41	50	47	46	49	61	60	63	54	47
LAB-12 (COLOR)	35	34	33	32	33	31	32	32	37	36
LAB-14 (COLOR)	54	54	52	54	54	62	52	56	58	54
LAB-23 (COLOR)	42	42	51	48	47	48	50	42	48	47
	41	47								
LAB-25 (COLOR)	40	30	40	40	30	30	40	30	40	
LAB-35 (COLOR)	48	46	48	48	48	39	48	48	48	48
LAB-40 (COLOR)	29	31	32	30	31	31	32	32	30	30
*LAB-46 (ES)	70	55	60	100	75					
LAB-27 (NAA)	44	42	44	44	44	50	45	46	45	43
	46	44								

* 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; ELECTR - electrogravimetry; FA - fire assay with gravimetric finish; FA-AA - fire assay with atomic absorption finish; FA-ES - fire assay with emission spectroscopic finish; NAA - neutron activation analysis; COMB - combustion; XRF - X-ray fluorescence.

TABLE 8 (cont'd)

Au (µg/g)

LAB- 2 (AA)	7.2	7.3	7.5	7.0	7.1	7.0	7.3	6.9	7.2	6.9
LAB- 6 (AA)	7.3	7.3	7.3	7.2	6.7	6.9	7.0	6.9	7.0	7.3
LAB- 9 (AA)	8.8	8.6	8.6	8.7	8.6	8.9	8.7	8.8	8.7	8.5
LAB-20 (AA)	8.0	7.6	7.6	7.5	8.0	7.5	7.5	8.0	7.5	8.0
LAB-21 (AA)	8.1	8.2	8.5	8.2						
LAB-35 (AA)	8.5	8.3	8.2	8.4	8.5	8.3	8.4	8.5	8.5	8.4
LAB- 5 (FA)	8.2	8.4	8.6	8.4	8.0	8.0	8.0	8.0	8.2	8.4
LAB-10 (FA)	8.1	7.8	7.4	8.1	7.2	7.9	7.8	7.7	7.4	7.9
LAB-12 (FA)	7.6	7.8	7.8	7.5	7.8	7.5	7.7	7.5	7.6	7.6
LAB-14 (FA)	6.9	6.9	7.5	6.5	6.9	7.5	8.2	8.2	8.2	8.2
LAB-19 (FA)	7.3	7.5	7.3	7.5	7.4	7.3	7.1	7.4	7.7	7.5
LAB-25 (FA)	7.4	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.4	7.5
LAB-27 (FA)	6.9	6.9	6.7	6.7	7.0	6.8	6.7	6.7	6.9	6.9
LAB-40 (FA)	7.2	7.4								
*LAB-41 (FA)	6.2	5.9								
LAB-43 (FA)	7.0	7.0	6.7	6.7	6.8	7.0	7.0	6.7	6.8	6.8
LAB- 2 (FA-AA)	6.5	6.6	6.7	6.9	6.4	7.0	6.9	6.9	7.1	6.9
LAB-21 (FA-AA)	7.5	7.5	7.6	7.6	7.4	7.3	7.5	7.7	7.5	7.2
LAB-29 (FA-AA)	7.3	7.2	7.1	7.1	7.6	7.6	7.4	7.4	7.4	7.8
LAB-17 (FA-ES)	7.4	7.3	6.8	7.2	7.4	7.2	7.1	7.0	7.3	7.5
LAB-44 (FA-ES)	7.2	6.0	7.0	3.5*	7.5	7.2	8.0	8.5	6.8	6.5

Bi (µg/g)

LAB-50 (AA)	31	18	21	17	19	22	23	24		
LAB- 3 (COLOR)	32	33	35	34	33	31	31	33	32	31
LAB-46 (ES)	22	25	20	20	15					

CaO (wt %)

LAB- 5 (AA)	.083	.083	.084	.084	.087	.083	.084	.085	.084	.088
* LAB-10 (AA)	.168	.168	.168	.154	.154	.140	.140	.140	.154	.154
LAB-12 (AA)	.090	.088	.087	.087	.088	.087	.087	.088	.088	.088
* LAB-14 (AA)	.042	.042	.042	.042	.042	.042	.042	.042	.042	.042
LAB-17 (AA)	.102	.098	.101	.098	.102	.101	.102	.101	.102	.101
LAB-19 (AA)	.085	.090	.090	.091	.095	.094	.085	.085	.097	.085
LAB-23 (AA)	.084	.084	.084	.084	.084	.098	.098	.084	.098	.098
	.084	.084	.084	.084	.084	.098	.098	.098	.098	.098
LAB-24 (AA)	.094	.091	.092	.091	.088	.092	.091	.091	.088	.087
LAB-25 (AA)	.084	.084	.084	.084	.084	.084	.084	.084	.084	.084
LAB-26 (AA)	.099	.104	.101	.105	.101	.102	.106	.104	.099	.106
LAB-35 (AA)	.107	.107	.107	.107	.107	.104	.104	.107	.107	.104
LAB-37 (AA)	.050	.063	.057	.060	.073	.055	.063	.060	.049	.050
LAB-40 (AA)	.113	.110	.112	.109	.113	.111	.110	.110	.113	.111
* LAB-41 (AA)	.084	.077	.074	.078						
LAB-44 (AA)	.111	.105	.095	.102	.091	.092	.087	.087	.087	.099
LAB-50 (AA)	.098	.084	.112	.098	.098	.098	.112	.098	.098	.098
* LAB- 6 (TITR)	.854	.728	.728	.728	.728	.965	.840	.714	.840	.714

Cd (µg/g)

LAB-23 (AA)	110	110	110	110	110	120	120	120	120	120
	110	110	120	120	110	120	120	120	120	120
LAB-46 (ES)	75	60	90	100	70					

TABLE 8 (cont'd)

Cu (wt %)

LAB- 7 (AA)	24.77	24.44	25.19	25.09	24.50	24.45	24.76	24.96	24.60	24.64
	25.05	24.62	24.96	24.69	24.65	24.92	25.24	25.11	25.21	25.09
LAB-20 (AA)	24.37	24.78	24.62	24.35	24.64	24.31	24.55	24.49	24.91	24.26
LAB-26 (AA)	24.95	24.97	24.87	25.00	24.87	24.86	24.90	24.87	24.99	24.86
* LAB-29 (AA)	24.40	24.40	24.60	25.50 *	24.40	24.30	24.20	24.40	24.40	24.30
* LAB-39 (AA)	25.40	25.32	25.21	25.23	25.29	25.29	25.25	25.29	25.32	25.36
LAB- 1 (TITR)	24.91	24.74	24.76	24.74	24.70	24.76	24.78	24.76	24.68	24.73
	24.65	24.71	24.82	24.71	24.67	24.65	24.64	24.82	24.77	24.63
	24.65	24.65	24.78	24.74	24.73	24.72	24.84	24.75	24.72	24.70
LAB- 4 (TITR)	24.75	24.75	24.80	24.72	24.75	24.80	24.70	24.70	24.72	24.72
LAB- 5 (TITR)	24.54	24.62	24.58	24.61	24.56	24.58	24.52	24.55	24.58	24.56
* LAB- 9 (TITR)	23.43	23.51	23.44	23.44	23.44	23.73	23.70	23.77	23.70	23.70
LAB-12 (TITR)	24.64	24.76	24.67	24.80	24.88	24.70	24.75	24.70	24.62	24.71
LAB-14 (TITR)	24.73	24.73	24.73	24.74	24.72	24.73	24.75	24.75	24.74	24.73
LAB-20 (TITR)	24.27	24.51	24.31	24.36	24.58	24.61	24.68	24.66	24.68	24.47
LAB-30 (TITR)	24.70	24.75	24.75	24.55	24.60	24.60	24.80	24.75	24.75	24.75
	24.70	24.75	24.60	24.60	24.60	24.75	24.80			
LAB-36 (TITR)	24.69	24.62	24.62	24.62	24.59	24.55	24.62	24.59	24.62	24.62
LAB-37 (TITR)	24.80	24.80	24.75	24.80	24.80	24.80	24.75	24.80	24.80	24.80
LAB-40 (TITR)	24.67	24.63	24.62	24.64	24.65	24.60	24.53	24.53	24.57	24.58
LAB-43 (TITR)	24.61	24.58	24.61	24.64	24.60	24.57	24.57	24.61	24.63	24.59
LAB-44 (TITR)	24.60	24.56	24.75	24.62	24.59	24.53	24.48	24.60	24.62	24.58
LAB-46 (TITR)	24.52	24.38	24.46	24.46	24.38					
* LAB-46 (TITR)	24.10	24.62	24.10	24.10	24.35					
LAB- 4 (ELECTR)	24.72	24.77	24.74	24.79	24.71					
LAB- 6 (ELECTR)	24.68	24.68	24.71	24.68	24.71	24.67	24.70	24.70	24.70	24.68
LAB-10 (ELECTR)	24.92	24.92	24.89	24.89	24.90	24.87	24.85	24.88	24.88	24.85
LAB-12 (ELECTR)	24.72	24.71	24.71	24.72	24.71	24.70	24.72	24.69	24.71	24.69
LAB-17 (ELECTR)	24.84	24.86	24.84	24.80	24.78	24.74	24.78	24.80	24.76	24.76
LAB-19 (ELECTR)	24.57	24.61	24.61	24.62	24.62	24.59	24.56	24.60	24.59	24.63
LAB-21 (ELECTR)	24.60	24.57	24.57	24.56						
LAB-21 (ELECTR)	24.72	24.71	24.75	24.75	24.73	24.69	24.67	24.67	24.73	24.69
LAB-21 (ELECTR)	24.79	24.76	24.64	24.62						
LAB-21 (ELECTR)	24.62	24.62	24.65	24.62	24.63	24.64	24.59	24.59	24.66	24.65
LAB-23 (ELECTR)	24.66	24.67	24.66	24.66	24.66	24.66	24.66	24.67	24.67	24.66
LAB-25 (ELECTR)	24.78	24.74	24.74	24.71	24.76	24.74	24.71	24.72	24.69	24.74
LAB-27 (ELECTR)	24.92	24.90	24.95	24.93	24.95	24.98	24.97	24.92	24.92	24.96
LAB-30 (ELECTR)	24.72	24.62	24.71	24.72	24.63	24.60	24.65	24.63	24.64	24.65
	24.67									
LAB-32 (ELECTR)	24.86	24.88	24.80	24.75	24.87	24.74	24.80	24.75	24.84	24.84
LAB-35 (ELECTR)	24.79	24.79	24.74	24.77	24.74	24.81	24.83	24.83	24.82	24.79
LAB-38 (ELECTR)	24.66	24.70	24.67	24.71	24.60	24.72	24.73	24.74	24.68	24.85
LAB-41 (ELECTR)	24.68	24.53	24.63	24.73	24.56	24.64	24.62	24.65	24.63	24.64
	24.62	24.62								
LAB-50 (ELECTR)	25.00	25.04	25.03	24.94	24.92	25.09	25.06	25.05	25.00	24.94

Fe (wt %)

LAB-30 (TITR)	30.94	30.99	30.89	30.99	30.78	30.94				
LAB-30 (TITR)	30.99	30.89	30.79	30.94						
LAB-35 (TITR)	30.89	30.85	30.87	30.80	30.81	30.81	30.79	30.78	30.76	30.76
LAB-36 (TITR)	30.60	30.60	30.62	30.61	30.60	30.60	30.61	30.56	30.61	30.60
* LAB-46 (TITR)	31.36	31.22	31.29	31.45	31.45					
LAB-46 (TITR)	30.87	30.94	30.94	30.87	30.53*					

Hg (μg/g)

LAB- 4 (AA)	63	67	67	67	64	62	60	62	62	64
LAB- 5 (AA)	60	62	60	59	60	61	59	59	59	61
LAB- 6 (AA)	62	62	60	61	58	63	61	63	63	63
LAB-12 (AA)	67	66	64	62	63	64	64	63	65	64
LAB-14 (AA)	64	67	64	64	70	68	68	66	66	64
LAB-19 (AA)	55	55	54	55	54	55	53	53	54	54
LAB-20 (AA)	60	67	61	64	57	63	69	69	65	69
*LAB-23 (AA)	84	80	83	87	83	80	76	80	82	78
LAB-25 (AA)	63	63	64	62	64	62	64	61	61	61
LAB-26 (AA)	67	65	64	64	65	63	67	66	65	64
*LAB-27 (AA)	96	96	92	90	92	90	80	94	86	74
LAB-29 (AA)	58	57	59	58	59	60	60	60	58	59
LAB-37 (AA)	60	60	59	59	61	59	59	59	58	60
LAB-44 (AA)	53	54	51	51	56	53	52	54	56	59
LAB-40 (COLOR)	59	60	60	63	62	60	57	59	60	59
*LAB-46 (ES)	90	60	80	110	70					

TABLE 8 (cont'd)

MgO (wt %)

LAB- 5 (AA)	1.05	1.05	1.03	1.04	1.04	1.04	1.03	1.04	1.04	1.04
LAB-10 (AA)	1.01	1.01	1.03	1.03	1.03	1.01	1.03	1.03	1.01	1.03
LAB-12 (AA)	1.02	1.02	1.02	1.02	1.02	1.02	1.02	1.03	1.02	1.02
LAB-14 (AA)	1.28	1.28	1.31	1.29	1.31	1.31	1.31	1.26	1.29	1.29
LAB-17 (AA)	1.11	1.10	1.11	1.09	1.10	1.10	1.10	1.10	1.08	
LAB-19 (AA)	1.13	1.12	1.14	1.12	1.12	1.10	1.10	1.12	1.10	1.10
LAB-23 (AA)	1.16	1.16	1.14	1.16	1.16	1.16	1.19	1.23	1.16	1.19
	1.16	1.16	1.16	1.16	1.16	1.23	1.23	1.19	1.19	1.19
LAB-24 (AA)	1.18	1.18	1.18	1.17	1.18	1.17	1.17	1.17	1.17	1.17
LAB-25 (AA)	1.23	1.23	1.23	1.19	1.19	1.23	1.19	1.23	1.23	1.23
LAB-26 (AA)	1.10	1.09	1.10	1.09	1.10	1.08	1.10	1.08	1.09	1.08
LAB-35 (AA)	1.13	1.13	1.13	1.10	1.13	1.13	1.10	1.10	1.10	1.10
* LAB-37 (AA)	.11	.12	.11	.11	.12	.11	.11	.11	.11	.11
LAB-40 (AA)	1.04	1.02	1.03	1.02	1.03	1.03	1.02	1.02	1.04	1.04
* LAB-41 (AA)	.18	.20	.17	.19						
LAB-44 (AA)	.99	1.06	.91	.99	.96	1.14	1.01	1.06	1.04	.96
LAB-50 (AA)	1.03	.99	.99	1.06	1.04	1.03	1.08	1.06	1.04	1.09
* LAB- 6 (TITR)	.59	.67	.59	.67	.59	.59	.59	.67	.67	.67

Mo (μg/g)

LAB-35 (AA)	40	40	60	40	40	60	40	40	20	20
LAB-46 (ES)	25	25	20	20	30					

Pb (wt %)

LAB- 5 (AA)	.114	.109	.110	.104	.099	.112	.099	.101	.094	.102
LAB- 6 (AA)	.100	.100	.100	.100	.100	.100	.100	.100	.100	.100
LAB- 7 (AA)	.125	.136	.135	.125	.137	.123	.134	.125	.134	.137
	.123	.122	.131	.127	.124	.122	.124	.123	.134	.125
LAB- 9 (AA)	.090	.091	.091	.090	.090	.089	.091	.089	.090	.091
LAB-10 (AA)	.110	.110	.110	.110	.110	.100	.100	.100	.100	.100
LAB-12 (AA)	.102	.101	.101	.103	.101	.101	.101	.101	.101	.100
LAB-14 (AA)	.130	.130	.130	.130	.130	.130	.130	.130	.130	.130
LAB-19 (AA)	.103	.102	.103	.105	.105	.103	.105	.105	.106	.106
LAB-20 (AA)	.107	.108	.111	.108	.108	.114	.112	.111	.115	.111
LAB-21 (AA)	.096	.095	.096	.097	.095	.098	.096	.095	.100	.100
LAB-21 (AA)	.132	.132	.128	.132	.130	.128				
LAB-21 (AA)	.110	.110	.110	.100	.090	.100	.090	.090	.090	.100
LAB-23 (AA)	.120	.120	.120	.120	.120	.110	.160	.100	.100	.110
	.130	.130	.120	.160	.120	.130	.110	.130	.110	.110
LAB-25 (AA)	.100	.100	.100	.100	.100	.100	.100	.100	.100	.100
LAB-26 (AA)	.107	.108	.109	.109	.109	.108	.107	.109	.108	.109
LAB-27 (AA)	.110	.112	.109	.110	.110	.112	.112	.112	.112	.112
LAB-29 (AA)	.107	.105	.098	.106	.102	.105	.105	.098	.106	.099
LAB-35 (AA)	.096	.096	.094	.092	.094	.092	.094	.096	.094	.096
LAB-37 (AA)	.102	.099	.100	.100	.102	.102	.102	.103	.103	.105
LAB-38 (AA)	.095	.100	.100	.100	.100	.100	.100	.100	.100	.100
LAB-39 (AA)	.117	.119	.118	.115	.111	.114	.115	.115	.114	.114
LAB-40 (AA)	.108	.108	.107	.107	.106	.107	.107	.107	.106	.106
LAB-41 (AA)	.088	.089	.089	.094	.090	.091	.087	.090	.090	.090
	.088	.090								
LAB-41 (AA)	.094	.090	.091	.090	.088	.090				
LAB-44 (AA)	.107	.105	.107	.103	.107	.103	.103	.104	.106	.107
LAB-50 (AA)	.112	.108	.108	.106	.106	.106	.106	.106	.108	.108
LAB- 1 (TITR)	.110	.110	.100	.110	.110	.110	.110	.100	.100	.110
LAB-36 (GRAV)	.080	.080	.080	.090	.100	.090	.080	.090	.090	.100
LAB-46 (GRAV)	.100	.090	.100	.080	.150*					
*LAB-46 (GRAV)	.210	.190	.200	.200	.210					

S (wt %)

LAB-35 (GRAV)	35.63	35.65	35.65	35.57	35.58	35.71	35.77	35.61	35.72	35.72
LAB-44 (GRAV)	35.70	35.63	35.72	35.61	35.61	35.66	35.72	35.70	35.73	35.71
LAB-46 (GRAV)	35.12	35.90	35.40	35.56	35.60					
LAB-46 (GRAV)	35.55	34.64	35.50	35.19	35.73					
*LAB-19 (COMB)	36.90	36.70	36.60	36.75	36.80	36.70	36.95	37.20	37.10	36.80

TABLE 8 (cont'd)

Se (μg/g)

LAB-27 (AA)	139	140	154	147	144	136	133	150	145	137
LAB-40 (AA)	118	115	114	113	115	117	115	114	114	113
LAB-50 (AA)	117	117	129	125	129	121	125	131		
LAB- 1 (COLOR)	105	100	111	104	104	98	106	104	106	108
LAB- 3 (COLOR)	103	105	110	107	100	96	110	110	117	99
LAB-14 (COLOR)	140	120	130	130	120	110	110	120	120	130
LAB-23 (COLOR)	129	126	126	131	127	131	131	127	126	126
LAB- 6 (XRF)	127	124	136	129	130	124	126	126	126	127
* LAB-25 (XRF)	60	70	60	60	70	70	60	70	60	70

SiO_2 (wt %)

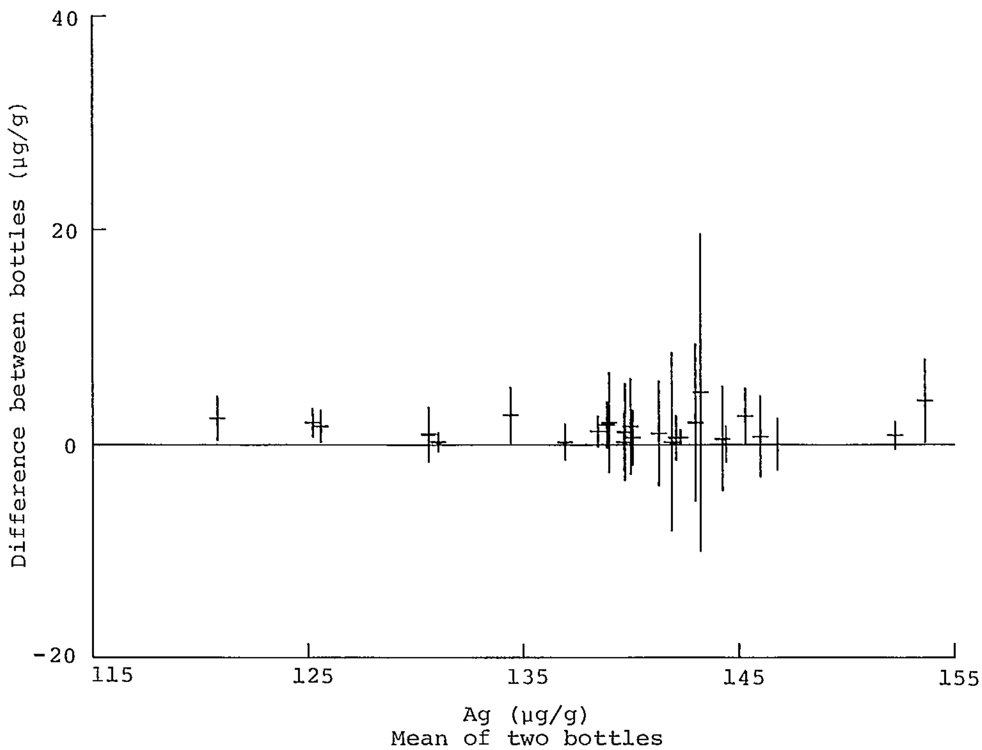
LAB-26 (AA)	2.65	2.61	2.61	2.65	2.65	2.65	2.65	2.61	2.65	2.65
LAB- 6 (GRAV)	2.74	2.70	2.67	2.70	2.67	2.78	2.80	2.80	2.70	2.74
*LAB-10 (GRAV)	.98	1.01	1.05	1.05	.98	.96	.96	.96	.96	.96
LAB-12 (GRAV)	2.61	2.57	2.57	2.61	2.63	2.52	2.57	2.61	2.59	2.59
LAB-14 (GRAV)	2.59	2.59	2.50	2.50	2.59	2.50	2.50	2.50	2.50	2.50
LAB-19 (GRAV)	2.63	2.63	2.63	2.61	2.63	2.65	2.61	2.65	2.63	2.70
LAB-23 (GRAV)	2.50	2.57	2.46	2.57	2.50	2.52	2.57	2.52	2.46	2.55
LAB-24 (GRAV)	2.64	2.61	2.60	2.57	2.59	2.77	2.81	2.54	2.69	2.60
LAB-25 (GRAV)	2.78	2.76	2.74	2.80	2.85	2.76	2.74	2.76	2.76	2.78
LAB-27 (GRAV)	2.59	2.59	2.57	2.61	3.00*	2.61	2.57	2.61	2.46	2.57
LAB-35 (GRAV)	2.38	2.47	2.44	2.32	2.32	2.55	2.53	2.55	2.51	2.51
LAB-36 (GRAV)	2.55	2.55	2.59	2.59	2.63	2.55	2.55	2.50	2.59	2.63
LAB-40 (GRAV)	2.40	2.48	2.46	2.37	2.37	2.37	2.42	2.25	2.35	2.40
LAB-41 (GRAV)	2.61	2.63	2.59	2.61						
LAB-46 (GRAV)	3.06	3.02	2.95	2.91	2.95					
LAB-46 (GRAV)	2.82	2.78	2.78	2.78	2.63					
*LAB-50 (GRAV)	3.27	3.55	3.25	3.40	2.95	3.38	3.40	3.29	3.51	3.27

Te (μg/g)

LAB-27 (AA)	6	5	5	7	7	6	5	6	4	4
LAB-40 (AA)	23	23	23	23	22	23	23	23	21	23
LAB-50 (AA)	34	29	29	35	26	43	33	34		
LAB- 3 (COLOR)	19	20	18	20	20	21	19	19	18	18
LAB-23 (COLOR)	17	18	17	16	16	17	16	18	16	16
LAB- 6 (XRF)	17	18	17	17	17	16	16	15	16	16
LAB-25 (XRF)	30	30	30	30	20	30	20	30	20	20

Zn (wt %)

LAB- 5 (AA)	3.13	3.12	3.11	3.14	3.15	3.12	3.10	3.09	3.11	3.14
LAB- 6 (AA)	3.32	3.31	3.32	3.33	3.31	3.34	3.32	3.32	3.35	3.35
LAB- 7 (AA)	3.31	3.31	3.32	3.32	3.27	3.27	3.27	3.34	3.26	3.25
	3.27	3.28	3.22	3.30	3.30	3.28	3.32	3.31	3.36	3.32
LAB- 9 (AA)	3.17	3.21	3.15	3.18	3.14	3.21	3.18	3.18	3.13	3.15
LAB-10 (AA)	3.24	3.24	3.25	3.24	3.24	3.24	3.24	3.26	3.25	3.22
LAB-12 (AA)	3.19	3.18	3.13	3.12	3.12	3.15	3.17	3.18	3.19	3.15
LAB-14 (AA)	3.20	3.20	3.30	3.20	3.30	3.20	3.20	3.20	3.30	3.30
LAB-20 (AA)	3.06	3.10	3.11	3.10	3.11	3.08	3.08	3.06	3.08	3.06
LAB-21 (AA)	3.31	3.31	3.32	3.33	3.31	3.34	3.34	3.34	3.31	3.33
LAB-21 (AA)	3.45	3.41	3.41	3.43	3.41	3.49				
LAB-21 (AA)	3.19	3.21	3.24	3.23	3.22	3.24	3.23	3.20	3.23	3.21
* LAB-23 (AA)	3.30	3.30	3.30	3.20	3.40	3.40	3.50	3.50	3.50	3.40
	3.30	3.40	3.30	3.30	3.30	3.50	3.50	3.50	3.40	3.60
LAB-25 (AA)	3.12	3.04	3.12	3.12	3.04	3.12	3.12	3.04	3.12	3.04
LAB-26 (AA)	3.21	3.24	3.19	3.22	3.20	3.21	3.21	3.20	3.19	3.18
LAB-27 (AA)	3.33	3.30	3.30	3.35	3.32	3.32	3.33	3.37	3.33	3.35
LAB-29 (AA)	3.30	3.31	3.24	3.30	3.25	3.31	3.29	3.21	3.29	3.28
LAB-35 (AA)	3.29	3.35	3.33	3.33	3.29	3.17	3.21	3.19	3.33	3.21
LAB-38 (AA)	3.20	3.22	3.21	3.18	3.17	3.19	3.20	3.17	3.15	3.18
* LAB-39 (AA)	4.25	4.20	4.25	4.23	4.18	4.35*	4.20	4.25	4.23	4.23
LAB-40 (AA)	3.19	3.22	3.20	3.18	3.20	3.22	3.17	3.23	3.22	3.20
LAB-41 (AA)	2.93	3.03	3.12	3.22	3.05	3.11	3.08	3.10	2.93	3.05
	3.05	3.10								
LAB-44 (AA)	3.30	3.18	3.24	3.23	3.23	3.23	3.24	3.24	3.18	3.23
LAB- 1 (TITR)	3.28	3.28	3.29	3.30	3.29	3.29	3.29	3.30	3.29	3.29
LAB- 1 (TITR)	3.22	3.18	3.19	3.22	3.21	3.24	3.19	3.20	3.19	3.23
LAB-19 (TITR)	3.32	3.34	3.34	3.32	3.34	3.34	3.32	3.32	3.37	3.34
LAB-24 (TITR)	3.24	3.23	3.26	3.20	3.19	3.12	3.26	3.19	3.25	3.20
*LAB-27 (TITR)	3.55	3.50	3.52	3.52	3.53	3.56	3.54	3.52	3.48	3.55
LAB-36 (TITR)	3.13	3.16	3.11	3.11	3.16	3.11	3.11	3.12	3.15	3.13
*LAB-46 (TITR)	2.62	2.92	2.62	2.75	2.70					
*LAB-46 (TITR)	3.01	3.00	2.99	3.12	3.05					



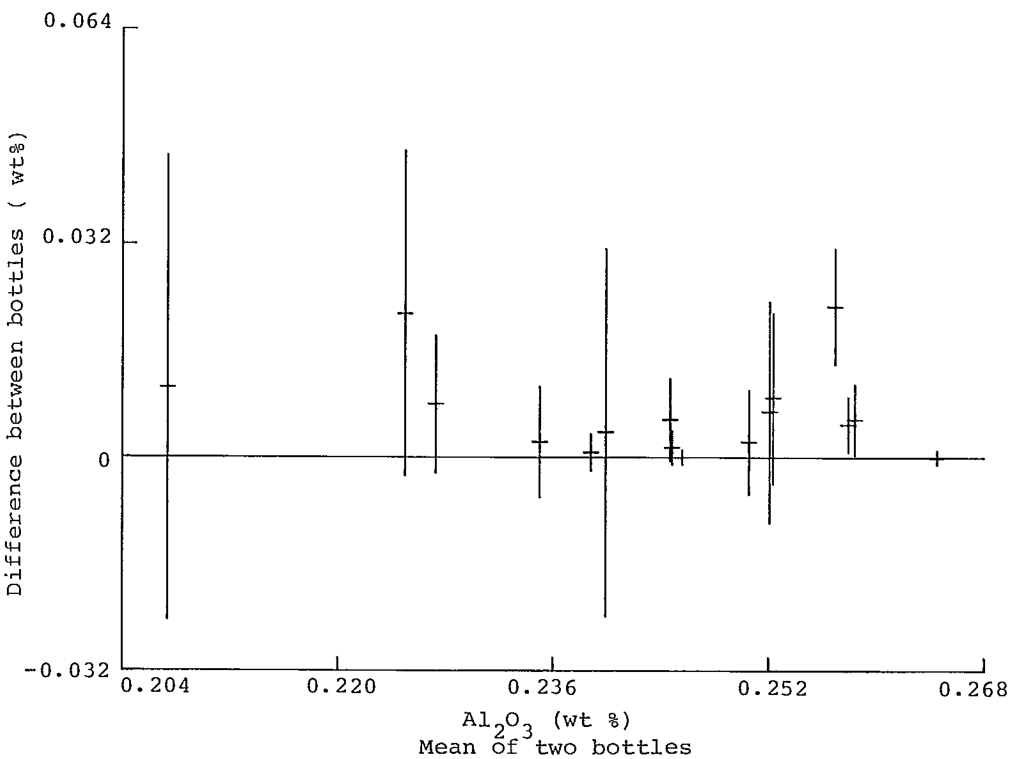
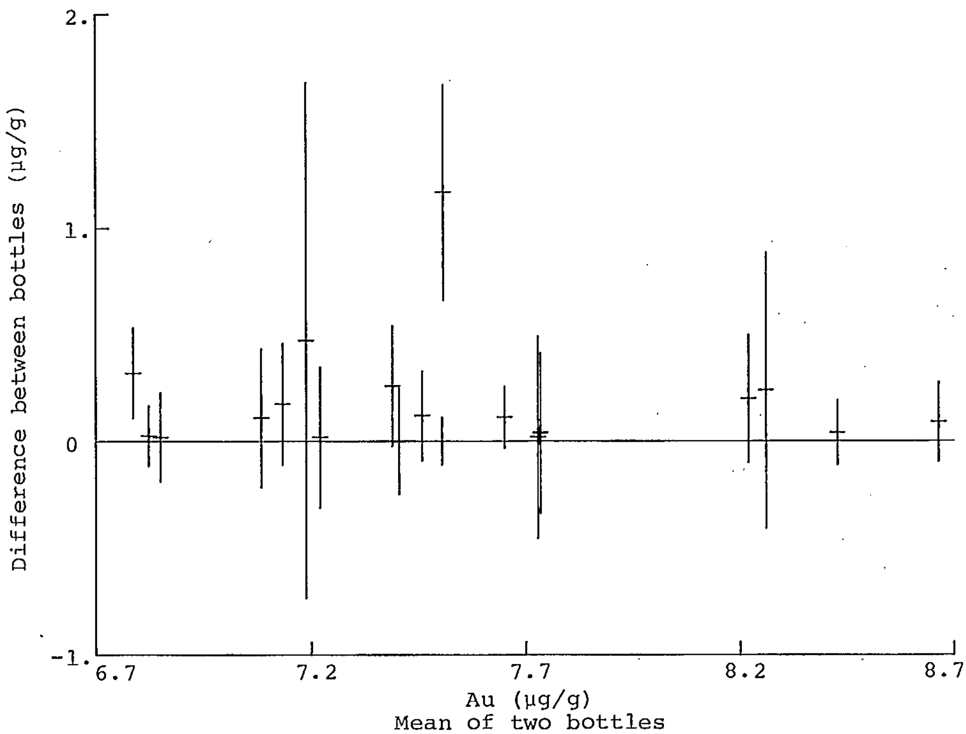


Fig. 1 - Degree of homogeneity of CCU-1. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.


2.
1.
0
-1.
Difference between bottles (μg/g)
6.7
7.2
7.7
8.2
8.7
Au (μg/g)
Mean of two bottles

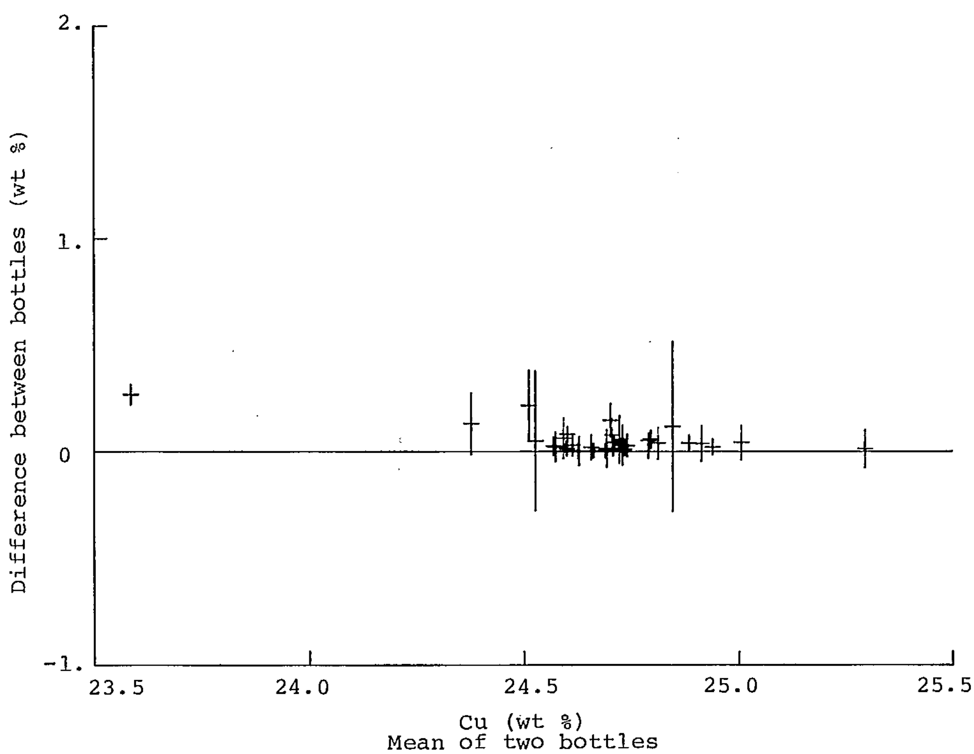
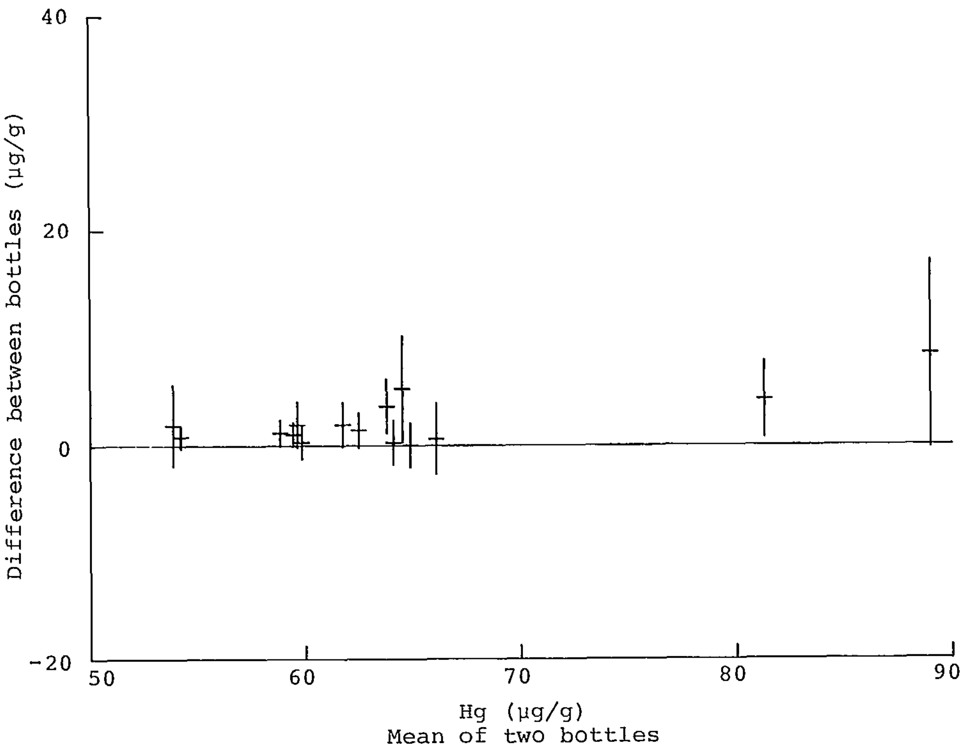

2.
1.
0
-1.
Difference between bottles (wt %)
23.5
24.0
24.5
25.0
25.5
Cu (wt %)
Mean of two bottles

Fig. 1 (cont'd)


40
20
0
-20
Difference between bottles (µg/g)
50
60
70
80
90
Hg (µg/g)
Mean of two bottles

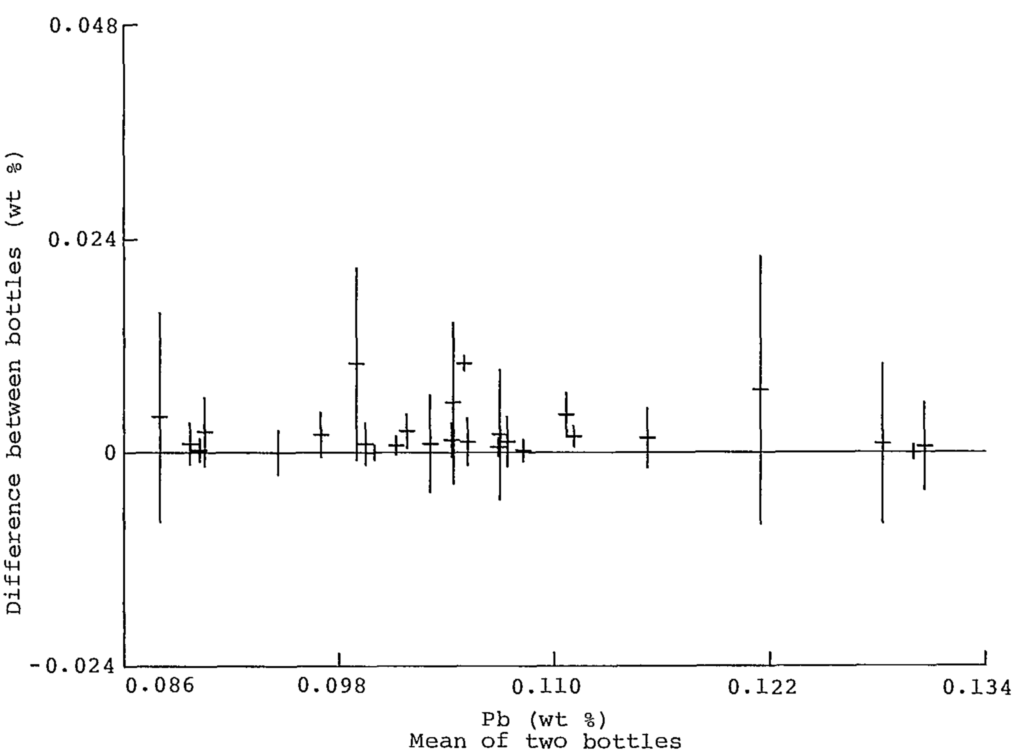
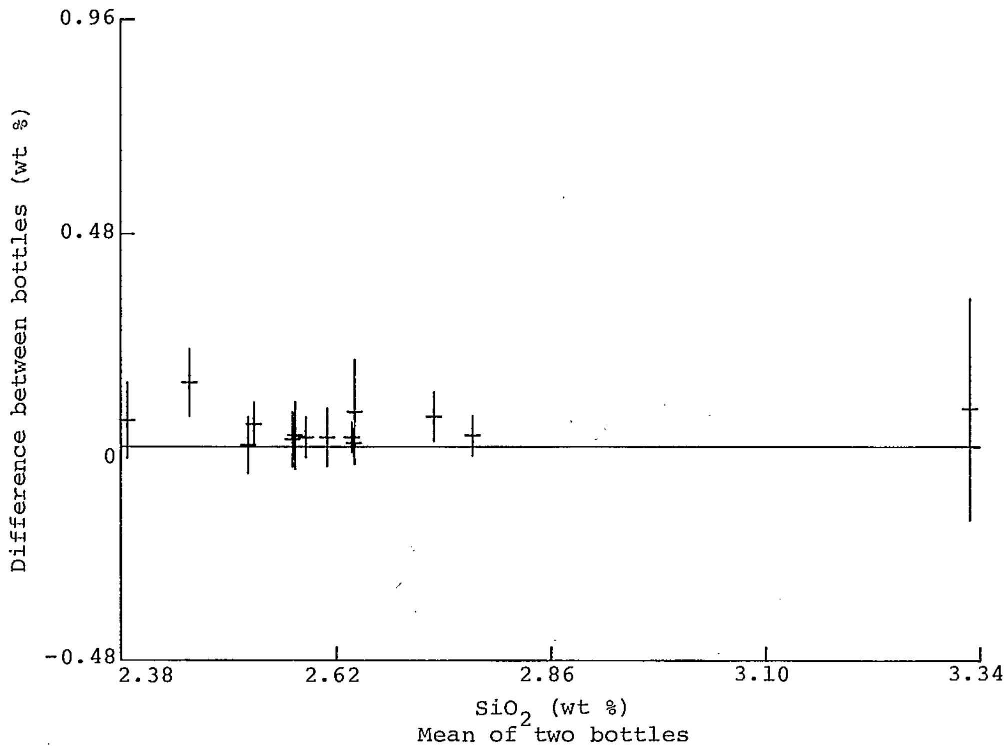

0.048
0.024
0
-0.024
Difference between bottles (wt %)
0.086
0.098
0.110
0.122
0.134
Pb (wt %)
Mean of two bottles

Fig. 1 (cont'd)


0.96
0.48
0
-0.48
Difference between bottles (wt %)
2.38
2.62
2.86
3.10
3.34
SiO_2 (wt %)
Mean of two bottles

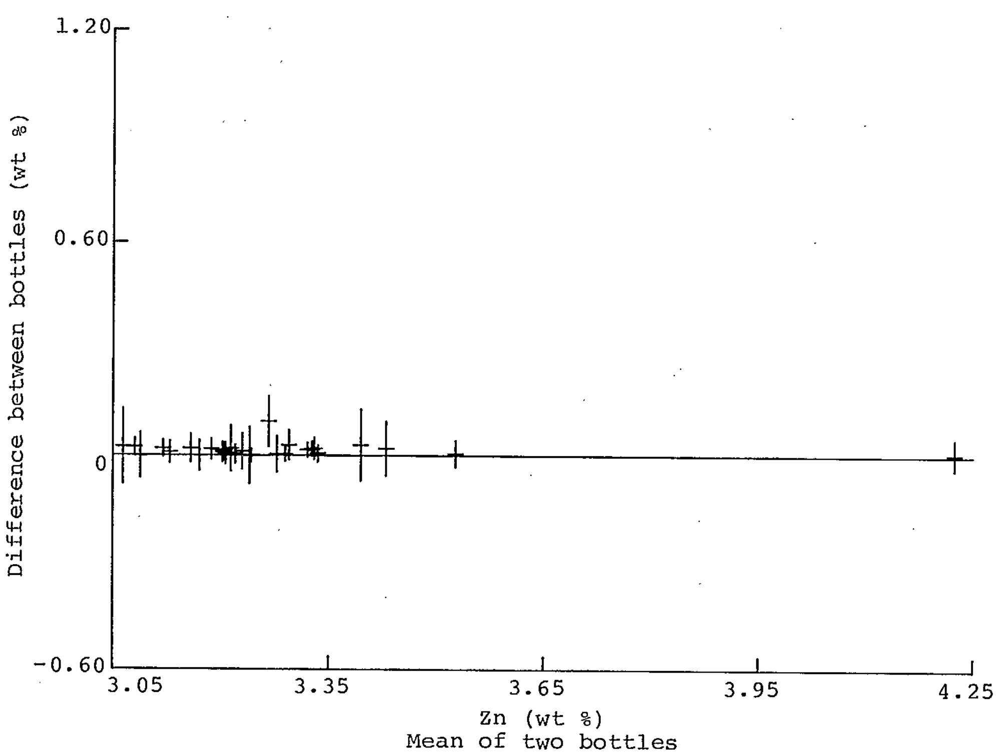

1.20
0.60
0
-0.60
Difference between bottles (wt %)
3.05
3.35
3.65
3.95
4.25
Zn (wt %)
Mean of two bottles

Fig. 1 (cont'd)

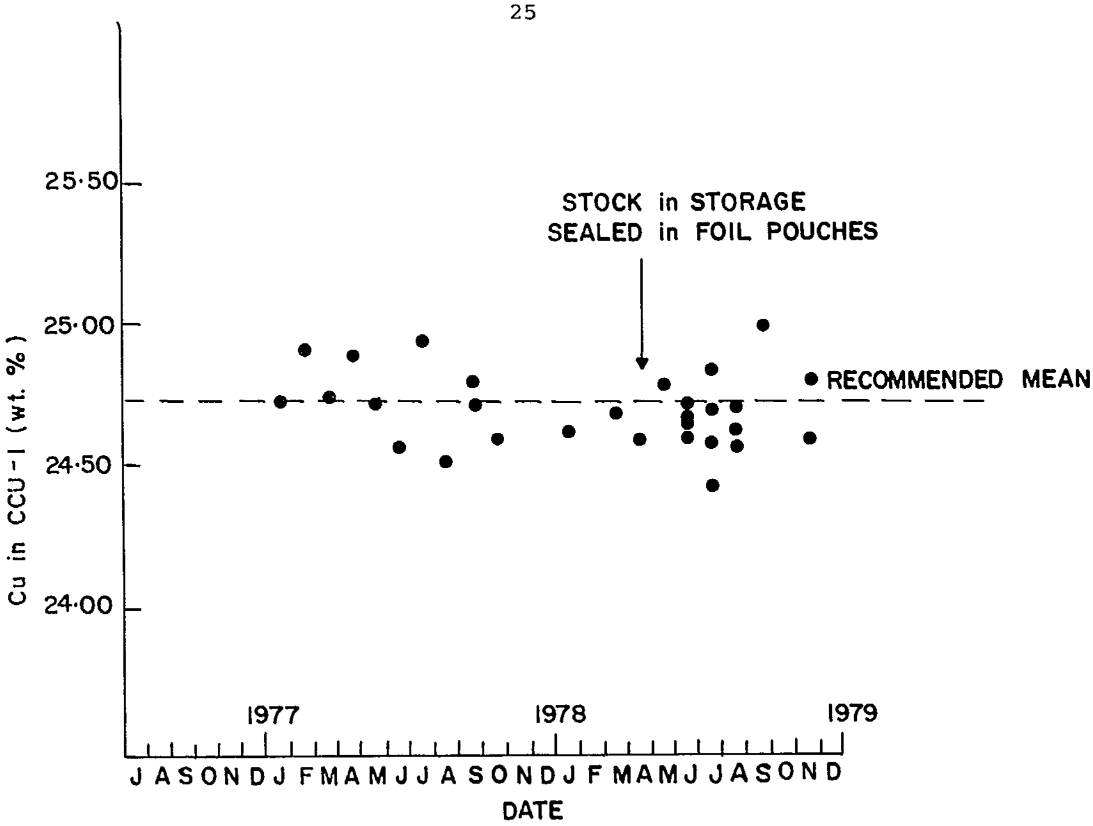


Fig. 2 - Laboratory means for Cu vs date of analysis

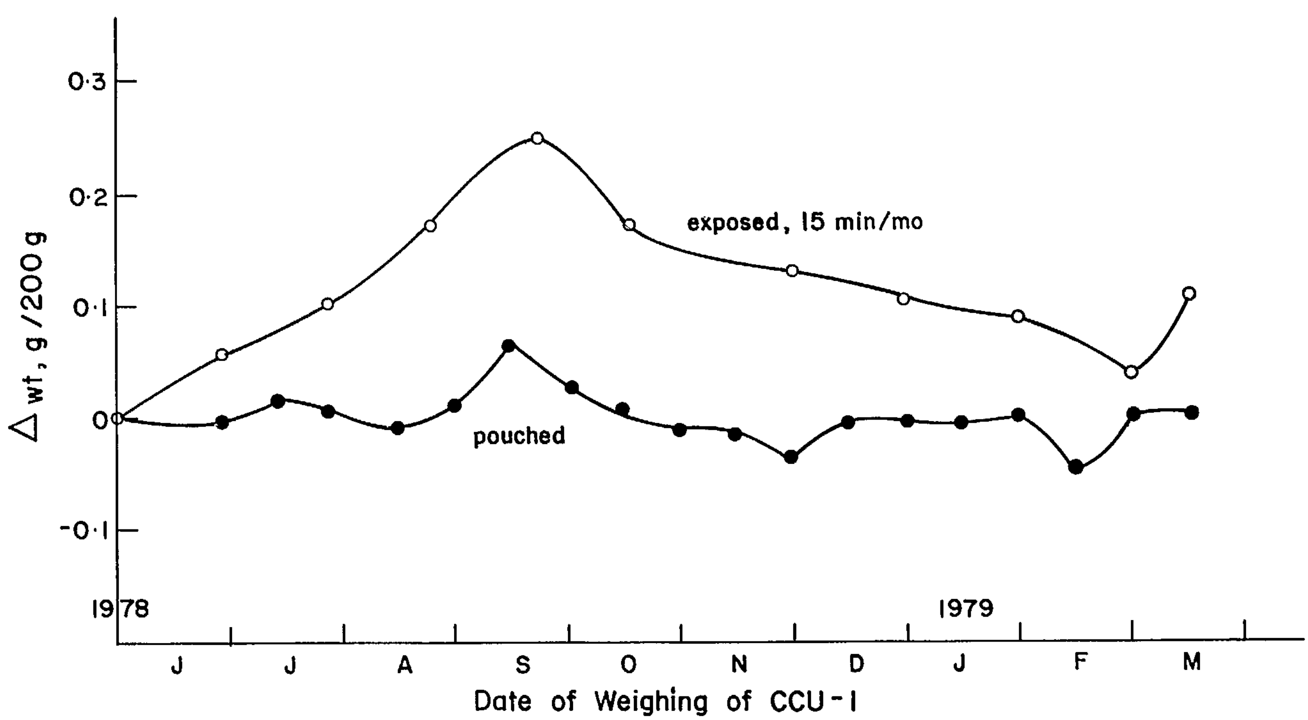


Fig. 3 - Change in weight of pouched and unpouched samples of CCU-1 vs storage time

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.

Brenda Mines Ltd., Peachland, British Columbia.

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

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

Centro De Investigacion Minera y Metalurgica, Santiago, Chile.

Chemex Labs Ltd., North Vancouver, British Columbia.

Cominco Ltd., Trail, British Columbia.

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

Falconbridge Copper Ltd., Opemiska Division, Abitibi, 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 Malayasia, Ipoh, Perab, Malayasia.

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.

Kamloops Research and Assay Laboratory Ltd., Kamloops, British Columbia.

LKAB Prospektering AB, Geochemical Laboratory, Stockholm, Sweden.

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

Newmont Mines Ltd., Similkameen Division, Princeton, British Columbia.

National Institute for Metallurgy, Randburg, South Africa.

Nchanga Consolidated Copper Mines Ltd., Kalulushi, Zambia.

Noranda Mines Ltd., Noranda, Quebec.

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.

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

University of Regina, Dept. of Geological Sciences, Regina, Saskatchewan.

APPENDIX B

OUTLINE OF PRINCIPAL TITRIMETRIC METHODS USED FOR COPPER IN CCU-1

The titrimetric methods for copper 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.

Short iodide (Laboratories 1, 5, 12, 20, 30, 38, 43, 46)

After sample decomposition, the acid insoluble material was removed by filtration and the filtrate was treated with bromine water to completely oxidize arsenic and antimony. The solution was neutralized with ammonium hydroxide and re-acidified with acetic acid, and iron was complexed with ammonium bifluoride. After the addition of potassium iodide, the liberated iodine was titrated with sodium thiosulphate solution.

Long iodide (Laboratories 4, 14, 36, 37, 40)

After sample decomposition, copper and other elements of the copper and arsenic groups were precipitated as sulphides from a dilute hydrochloric acid medium and thereby separated from iron, manganese and zinc. The mixed-sulphide precipitate was dissolved in nitric acid-bromine water and the solution was taken to fumes of sulphuric acid. The solution was ultimately neutralized with ammonium hydroxide and re-acidified with acetic acid. Potassium iodide was added and the liberated iodine was titrated with sodium thiosulphate solution.