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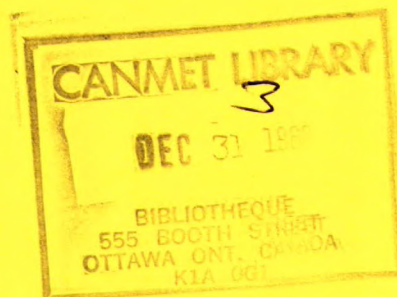
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SU-1a: A CERTIFIED NICKEL-COPPER-COBALT REFERENCE ORE

H.F. STEGER AND W.S. BOWMAN



MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES



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SU-1a: A CERTIFIED NICKEL-COPPER-COBALT REFERENCE ORE

by

H.F. Steger* and W.S. Bowman**

SYNOPSIS

A 332-kg sample of nickel-copper-cobalt ore, SU-1a, from the Sudbury region, Ontario, was prepared as a compositional reference material to replace the similar certified ore, SU-1, of which the stock had been exhausted. SU-1a was ground to minus 74 μm , blended in one lot, tested for homogeneity by X-ray fluorescence and chemical methods and bottled in 200-g units.

In a "free-choice" analytical program, 23 laboratories contributed results for one or more of nickel, copper, cobalt, platinum, palladium and silver in each of two bottles of SU-1a. Based on a statistical analysis of the data, the following recommended values were assigned: Ni, 1.233%; Cu, 0.967%; Co, 0.041%; Pt, 0.41 $\mu\text{g/g}$; Pd, 0.37 $\mu\text{g/g}$; and Ag, 4.3 $\mu\text{g/g}$.

In addition, values are reported for gold, iridium, rhodium, osmium and ruthenium for information purposes.

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Note: Major contributions to the certification of SU-1a were also made by other staff members of the Mineral Sciences Laboratories.

SU-1a: MINERAI DE REFERENCE CERTIFIE DE NICKEL-CUIVRE-COBALT

par :

H.F. Steger* et W.S. Bowman**

SYNOPSIS

Un échantillon de 332 kg de minerai de nickel-cuivre-cobalt, SU-1a, provenant de la région de Sudbury (Ontario) a été préparé comme matériau de référence de composition pour remplacer le minerai certifié analogue, SU-1, dont l'inventaire était épuisé. Le SU-1a a été broyé à une granulométrie de moins 74 μm , mélangé en lot, soumis à des essais d'homogénéité par la méthode de fluorescence des rayons X et autres méthodes chimiques et embouteillé en unités de 200 g.

En vertu d'un programme analytique de "libre choix", 23 laboratoires ont soumis les résultats sur chacun des deux flacons de SU-1a pour un ou plusieurs des éléments suivants: nickel, cuivre, cobalt, platine, palladium et argent. Suite à l'analyse statistique des données, les valeurs recommandées suivantes ont été assignées: Ni: 1,233%; Cu: 0,967%; Co: 0,041%; Pt: 0,41 $\mu\text{g/g}$; Pd: 0,37 $\mu\text{g/g}$; et Ag: 4,3 $\mu\text{g/g}$.

De plus, des valeurs ont été déterminées pour l'or, l'iridium, le rhodium, l'osmium et le ruthénium à titre d'information.

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INTRODUCTION

The preparation, characterization and certification of the nickel-copper-cobalt ore SU-1a is another example of the continuing endeavour of the Canadian Certified Reference Materials Project (CCRMP) to provide compositional reference ores, concentrates and related products typical of Canadian deposits and generally unavailable from other sources. These were prepared for use in analytical laboratories associated with mining, metallurgy and the earth sciences. Other certified reference materials are described in a catalogue available from CANMET, Energy, Mines and Resources, Ottawa, Canada (1).

SU-1a was intended to replace SU-1 the supply of which was exhausted (2). SU-1 was certified in 1973 to fill the need for nickel-bearing reference materials. In addition to certifying SU-1a for nickel, copper and cobalt, as was done for SU-1, it was decided to certify it also for platinum, palladium and silver.

An interlaboratory program was conducted to obtain results for nickel, copper, cobalt, platinum, palladium and silver from twenty three commercial, industrial and government laboratories using analytical methods of their choice. The results should therefore be indicative of the current "state-of-the-art" of the analysis for these elements.

NATURE AND PREPARATION

The raw material for SU-1a was donated to CCRMP in June 1977 by Inco Limited at Copper Cliff, Ontario and was typical of the feed to Inco's Clarabelle mill. It consisted essentially of a siliceous matrix with minor amounts of pyrrhotite, pyrite and pentlandite.

The sample was dry-ground in March 1978 to pass a 74- μ m screen. The powdered ore weighing approximately 332 kg was tumbled in a 570-L conical blender for 7 h and sampled systematically for analysis for nickel, copper and cobalt by X-ray fluorescence and chemical methods. SU-1a was found to be sufficiently homogeneous to qualify as a reference material and was bottled in 200-g

units which were heat-sealed in polyester-aluminum foil-polyethylene pouches to prevent oxidation while in storage at CANMET. The approximate mineralogical and chemical composition and particle size analysis are given in Tables 1 to 3.

INTERLABORATORY PROGRAM FOR CERTIFICATION

The laboratories that participated in the certification program are listed in Appendix A. Each was assigned a code number which bore no relation to its alphabetical order.

Each laboratory was requested to contribute five replicate results for nickel, copper, cobalt, platinum, palladium and silver on each of two bottles of SU-1a by methods of their own choice and to report the results on an "as is" basis. Some laboratories however deviated from the request for 10 results for each element or contributed results for fewer than the six elements. Where a laboratory submitted results by more than one method, each set was considered statistically independent.

The recommended values for the six certified elements are presented in Table 4. Methodological, statistical and other analytical information is presented in Tables 5 to 7. Analytical information for gold, some secondary precious metals, and for an additional seven elements is reported in Tables 8 and 9.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

DETECTION OF OUTLIERS

Any sets of results obviously suspect for methodological reasons were rejected. Sets with unusually high variance were examined and any individual outlying results were deleted. In extreme cases, entire sets with high variance were rejected. Also, sets of results whose means differed by more than twice the overall standard deviation from the initial mean value were not used in subsequent computations to avoid possible biasing of the statistics. See the discussion of the silver results for the special treatment given to this element.

Table 1 - Mineralogical composition of SU-1a

Mineral	wt %
Chlorite	23
Quartz	16
Feldspar	15
Mica	12
Amphibole	12
Calcite	1
Siderite	1
Magnetite + sphalerite	10
Pyrrhotite	4.1
Pentlandite + trace pyrite	3.1
Chalcopyrite	2.8
	100

Table 2 - Chemical composition of typical Clarabelle mill feed*

Element	wt %
Cu	1.2
Ni	1.3
Co	0.04
Fe	20
S	10
SiO ₂	38
Al	5.0
Ca	3.5
Mg	3.0
Pb	0.008
Bi	0.0005
As	0.003
Se	0.002
Pt	0.5 ppm
Pd	0.6
Au	0.2
Ag	5.6
Rh	0.1
Ru	0.06
Ir	0.03

*Results provided by Inco Limited, Copper Cliff, Ontario.

Table 3 - Particle size analysis (wet screen)

Size of fraction (µm)	wt %*
-104 + 74	3
- 74 + 55	12
- 55 + 46	10
- 46 + 37	2
- 37	73

*Mean of duplicate determinations.

HOMOGENEITY TESTS USING INTERLABORATORY RESULTS

Table 6 gives the means and coefficients of variation of each set of results and also 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 designated by the code REJECT. For cobalt, the code ****R** denotes zero variance for that set of results. For the six elements, 12 sets out of 108 were rejected. A rejection rate of 11% was typical of previous CCRMP ore certification programs. It must be mentioned that, with the exception of Lab. 24 which found a between-bottle difference for both nickel and platinum, the other laboratories all reported a between-bottle difference for one element only.

The degree of homogeneity of SU-1a is also illustrated in Fig. 1 in which the difference between the means of the results for the two bottles was plotted against the overall mean of the results for both bottles for each set. The vertical bar represents the 95% confidence interval of the former. If the 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 estimate the consensus values and their variance. This approach considers the results of the described certification program to be only one sampling out of a universal set of results. The analytical data were assumed to fit the model (3).

Table 4 - Recommended values and associated statistical parameters (outliers excluded)

Element	No. of Laboratories	No. of Results	Overall Mean	95% CL		σ_A^*
				Low	High	
			(wt %)	(wt %)	(wt %)	
Ni	22	242	1.233	1.225	1.241	0.008
Cu	24	242	0.967	0.962	0.972	0.007
Co	20	200	0.041	0.040	0.042	0.001
			($\mu\text{g/g}$)	($\mu\text{g/g}$)	($\mu\text{g/g}$)	
Pt	9	85	0.41	0.35	0.47	0.04
Pd	9	80	0.37	0.35	0.40	0.02
Ag	10	100	4.3	4.1	4.6	0.2

*Average within-set standard deviation.

where $x_{ij} = \mu + y_i + e_{ij}$
 x_{ij} = the j^{th} result in set i ,
 μ = the true consensus value,
 y_i = the discrepancy between the mean of the results in set i ($\bar{x}_{i.}$) and μ , and
 e_{ij} = the discrepancy between x_{ij} and $\bar{x}_{i.}$

It is assumed 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 is 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 consensus value of the assumed model is estimated by the overall mean $\bar{x}_{..}$:

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

where n_i = the number of results in set i , and
 k = the number of sets.

The value of σ^2 is estimated by s_1^2 which is given by

$$s_1^2 = \frac{\sum_i \sum_j^k n_i (x_{ij} - \bar{x}_{i.})^2}{\sum_i n_i - k}$$

The value of ω^2 is estimated by

$$\omega^2 = (s_2^2 - s_1^2) / \frac{1}{k-1} \left(\sum_i^k n_i - \sum_i^k n_i^2 / \sum_i^k n_i \right)$$

where

$$s_2^2 = \frac{\sum_i^k n_i (\bar{x}_{i.} - \bar{x}_{..})^2}{k-1}$$

The variance of the overall mean is given by

$$V[\bar{x}_{..}] = \left(\frac{\sum_i^k n_i^2 / (\sum_i^k n_i)^2}{1/\sum_i^k n_i} \right) \omega^2 + \left(\frac{k}{1/\sum_i^k n_i} \right) \sigma^2$$

and the 95% confidence limits for $\bar{x}_{..}$ are

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

It should be noted that 95% confidence limits denote that if the certification program were performed 100 times, the overall mean in 95 would fall within the prescribed limits.

The average within-set standard deviation, σ_A , is a measure of the average within-bottle precision as determined by the analytical methods used. The implication exists therefore that a laboratory using a method of average or better reproducibility should obtain individual results for a given certified element with a precision that is at least comparable to the reported value of σ_A .

Table 5(a) - Summary of analytical methods for nickel (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} (wt %)
<u>Atomic absorption</u>	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution 2-10% v/v HCl	7	1a, 8, 9, 12a, 18, 19, 21	90	1.231
	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution 2-10% v/v HNO ₃	3	6, 16, 23	30	1.225
	HClO ₄ ; final solution is unknown dilute HClO ₄	1	14a	10	1.230
	HClO ₄ + HNO ₃ ; final solution is unspecified dilute HClO ₄ + HNO ₃	1	20a	10	1.235
	HNO ₃ + Br ₂ + HF + HClO ₄ ; final solution is unspecified dilute HClO ₄	1	31	10	1.226
	Aqua regia, HF; final solution is unspecified HNO ₃ + HCl + HF	1	24	10	1.215
	Na ₂ O ₂ fusion; final solution is 4% HNO ₃	1	28a	10	1.248
	Na ₂ O ₂ fusion; final solution is unspecified dilute HNO ₃ containing KNO ₃ a deionizing agent	1	28b	12	1.225
	<u>Colorimetric</u>	HNO ₃ , HF, HNO ₃ , HClO ₄ ; final solution is 0.5% HCl; nickel as dimethylglyoximate in ammoniacal citrate + KI ₃	1	1b	10
<u>Gravimetric</u>	HClO ₄ ; final solution is unspecified dilute HNO ₃ , H ₂ SO ₄ , citric acid; copper removed by electrolysis; iron oxidized with KClO ₃ ; nickel precipitated with dimethylglyoxime	1	14b	10	1.230
	HNO ₃ , KClO ₃ , H ₂ SO ₄ , HF; final solution unspecified dilute HCl; tartaric acid neutralized with NH ₄ OH; nickel precipitated with dimethylglyoxime	1	20b	10	1.2403
	HNO ₃ , HCl; residue fused with Na ₂ CO ₃ and treated with HF to remove SiO ₂ ; final solution is unspecified dilute HCl; tartaric acid made ammoniacal; nickel precipitated with dimethylglyoxime	1	3	10	1.254
<u>Titrimetric</u>	HNO ₃ , HCl, HClO ₄ , HF; final solution is unspecified dilute HClO ₄ ; nickel precipitated with dimethylglyoxime; filtered, redissolved and nickel titrated with EDTA	1	15	10	1.256
<u>X-ray fluorescence</u>	Potassium pyrosulphate discs	1	126	10	1.239

Table 5(b) - Summary of analytical methods for copper (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} (wt. %)
<u>Atomic absorption</u>	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution is 2-10% v/v HCl	6	1a, 9a, 12a, 18, 19, 21	60	0.965
	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution is 2-10% v/v HNO ₃	3	6, 16, 23	30	0.967
	HNO ₃ + Br ₂ + HF + HClO ₄ ; final solution is unspecified dilute HClO ₄	2	15, 31	20	0.960
	HClO ₄ ; final solution is unspecified dilute HClO ₄	1	14a	10	0.958
	HClO ₄ + HNO ₃ ; final solution is unspecified dilute HClO ₄ + HNO ₃	1	20	10	0.957
	Aqua regia + HF; final solution is dilute HNO ₃ + HCl + HF	1	24	10	0.967
	HClO ₄ + HF; final solution is dilute HClO ₄	1	33	10	0.974
	Acid decomposition; no details	1	17	10	0.992
	Na ₂ O ₂ fusion; final solution is 4% HNO ₃	1	28a	10	0.972
	Na ₂ O ₂ fusion; final solution is unspecified dilute HNO ₃ containing KNO ₃ as deionizing agent	1	28b	12	0.958
<u>Colorimetric</u>	HNO ₃ + HCl + HF + HClO ₄ ; final solution is 0.5% HCl; hydroxylamine + tartaric acid; copper-cuproine complex is extracted into n-amyl alcohol	1	1b	10	0.977
<u>Electrolytic</u>	HNO ₃ + Br ₂ + HCl + H ₂ SO ₄ ; Cu electroplated from dilute H ₂ SO ₄ containing tartrate and hydrazine hydrochloride	1	9b	10	0.978
	HClO ₄ ; final solution is dilute HClO ₄ + HCl; iron reduced with Na ₂ SO ₃ ; Cu precipitated with H ₂ S; redissolved and Cu electroplated from dilute H ₂ SO ₄ + HNO ₃	1	14b	10	0.972
	HClO ₄ ; Cu electroplated from dilute HClO ₄ + HNO ₃ containing citrate	1	14c	10	0.971
<u>X-ray fluorescence</u>	Potassium pyrosulphate discs	1	12b	10	0.965
	Hoechst microwax FA1 discs	1	34	10	0.967

Table 5(c) - Summary of analytical methods for cobalt (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} (wt %)
<u>Atomic absorption</u>	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution is 2-10% v/v HCl	6	1a, 9, 12a, 18, 19, 21	60	0.041
	HNO ₃ + one or more of HCl, HF, HClO ₄ ; final solution 2-10% v/v HNO ₃	2	6, 16	20	0.041
	HClO ₄ ; final solution is unspecified dilute HClO ₄	1	14	10	0.039
	HClO ₄ + one or more of HNO ₃ , Br ₂ , HF, HCl; final solution is unknown dilute HClO ₄	3	15, 31, 33	30	0.040
	HClO ₄ + HNO ₃ ; final solution is unspecified dilute HClO ₄ + HNO ₃	1	20	10	0.040
	Aqua regia + HF; final solution is unspecified dilute HNO ₃ + HCl + HF	1	24	10	0.040
	HClO ₄ + HNO ₃ + HF + HCl; final solution is 7.5% HCl + 2.5% HNO ₃	1	30	10	0.033
	Acid decomposition; no details	1	17	10	0.042
	Na ₂ O ₂ fusion; final solution is 4% HNO ₃	1	28	10	0.042
<u>Colorimetric</u>	HNO ₃ + HCl + HF + HClO ₄ ; final solution is 0.5% HCl; Co-thiocyanate complex extracted into iso-amyl alcohol-ether	1	16	10	0.028
<u>X-ray fluorescence</u>	Potassium pyrosulphate discs	1	12b	10	0.041
	Hoechst microwax FA1 discs	1	34	10	0.047

Table 5(d) - Summary of analytical methods for platinum (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} ($\mu\text{g/g}$)
<u>Fire assay-atomic absorption</u>	Pb button cupelled, dissolved in aqua regia for a.a.	1	6	9	0.30
	NiS button dissolved in HCl for a.a.	1	23	10	0.53
<u>Fire assay-colorimetric</u>	Sn button dissolved in HCl; Pt and Pd isolated by ion-exchange; Pd extracted as p-nitrosodimethylaniline complex in chloroform; Pt extracted as SnCl_2 complex in 5% tributylphosphate in n-hexane	1	5	10	0.50
<u>Fire assay-emission spectrography</u>	Pb button cupelled to 5-100 mg Ag bead for em. spec.	3	12, 19, 24	30	0.39
	Pb button cupelled to 5 mg Ag bead; dissolved in aqua regia	1	33	10	0.35
	Pb button by arrested cupellation technique; lead bead for em. spec.	1	15	10	0.44
<u>Fire assay-neutron activation analysis</u>	Ni button dissolved in HCl; residues irradiated for 3 min in Slowpoke reactor	1	26	6	0.41

Table 5(e) - Summary of analytical methods for palladium (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} ($\mu\text{g/g}$)
<u>Fire assay-atomic absorption</u>	Pb button cupelled; silver bead dissolved in aqua regia	1	6	10	0.36
	Pb button cupelled; gold bead dissolved in aqua regia; boiled to dryness; residue dissolved in 0.5% La - 0.25% 8-hydroxyquinidine	2	24, 18	14	0.38
	NiS button dissolved in HCl	1	23	10	0.33
<u>Fire assay-colorimetric</u>	Sn button dissolved in HCl; Pt and Pd isolated by ion-exchange; extracted as p-nitroso dimethylaniline complex in chloroform	1	5	10	0.35
<u>Fire assay-emission spectrography</u>	Pb button cupelled to 5-100 mg Ag bead for em. spec.	2	12, 19	20	0.36
	Pb button by arrested cupellation technique; lead bead for em. spec.	1	15	10	0.43
<u>Fire assay-neutron activation analysis</u>	Ni button dissolved HCl; residue irradiated for 3 min in Slowpoke reactor	1	26	6	0.38

Table 5(f) - Summary of analytical methods for silver (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	\bar{x} ($\mu\text{g/g}$)
<u>Atomic absorption</u>	HNO_3 + HF; final solution is unspecified dilute HNO_3	1	16	10	3.8
	HF + H_2SO_4 ; residue fused with Na_2O_2 and dissolved in dilute HNO_3 ; silver extracted into iso-octyl-mercaptoacetate and stripped into dilute HCl	1	28	10	4.3
	1% $\text{Hg}(\text{NO}_3)_2$ in 50% HNO_3 ; final solution is 25% HNO_3	1	33a	10	4.1
<u>Fire assay-atomic absorption</u>	Pb bead partially cupelled; dissolved in dilute HNO_3	2	5, 15	20	4.5
	Pb bead partially cupelled; dissolved in 10% HNO_3 ; Ag^{110} tracer carried with samples	1	19	10	4.3
	Pb button; dissolved in dilute HNO_3	1	12	10	4.0
	Pb button cupelled; 5 mg Au bead dissolved in aqua regia and boiled to dryness; silver taken up in 0.5% La - 0.25% 8-hydroxyquinoline	1	24	10	4.8
<u>Emission spectrographic</u>	Sample mixed with graphite and sodium carbonate	1	36	10	5.1
	No details given	1	33b	10	4.1

Table 6 - Laboratory means, coefficients of variation and summary of t-test
between bottle results for certified constituents

	Co (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	.0381	.0003	5	.0384	.0003	A	10	.0382	.0003	.83
LAB- 1 (COLOR)	5	.0387	.0008	5	.0383	.0013	A	10	.0385	.0011	2.73
LAB- 6 (AA)	5	.0420	.0000	5	.0420	.0000	A	10	.0420	.0000	.00
LAB- 8 (AA)	5	.0244	.0002	5	.0245	.0002	A	10	.0245	.0002	.82
LAB- 9 (AA)	5	.0412	.0011	5	.0420	.0000	A	10	.0416	.0008	2.03
LAB-12 (AA)	5	.0397	.0003	5	.0398	.0003	A	10	.0398	.0003	.66
LAB-12 (XRF)	5	.0406	.0068	5	.0416	.0048	A	10	.0411	.0056	13.59
LAB-14 (AA)	5	.0386	.0009	5	.0384	.0009	A	10	.0385	.0008	2.21
LAB-15 (AA)	5	.0400	0.0000	5	.0414	.0005	***R**	10	.0407	.0008	2.02
LAB-16 (AA)	5	.0389	.0003	5	.0390	.0002	A	10	.0390	.0003	.72
LAB-17 (AA)	5	.0420	.0000	5	.0420	.0000	A	10	.0420	.0000	.00
LAB-18 (AA)	5	.0438	.0019	5	.0424	.0021	A	10	.0431	.0020	4.70
LAB-19 (AA)	5	.0398	.0008	5	.0400	.0004	A	10	.0399	.0006	1.42
LAB-20 (AA)	5	.0400	0.0000	5	.0402	.0004	A	10	.0401	.0003	.79
LAB-21 (AA)	5	.0404	.0002	5	.0402	.0002	A	10	.0403	.0002	.55
LAB-23 (AA)	5	.0472	.0004	5	.0476	.0005	A	10	.0474	.0005	1.09
LAB-24 (AA)	5	.0398	.0006	5	.0396	.0008	A	10	.0397	.0007	1.78
LAB-28 (AA)	5	.0420	.0006	5	.0419	.0009	A	10	.0419	.0007	1.69
LAB-30 (AA)	5	.0320	0.0000	5	.0330	.0007	***R**	10	.0325	.0007	2.18
LAB-31 (AA)	5	.0392	.0005	5	.0397	.0003	A	10	.0395	.0005	1.24
LAB-33 (AA)	5	.0412	.0016	5	.0400	.0007	A	10	.0406	.0013	3.32
LAB-34 (XRF)	5	.0466	.0019	5	.0470	.0020	A	10	.0468	.0019	4.00

Variance between sets, between bottles and within bottles = 2.05×10^{-5} , 0. and 2.25×10^{-6} , respectively.

	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- 5 (FA-AA)	5	4.3000	.1971	5	4.2200	.1789	A	10	4.2600	.1776	4.17
LAB- 6 (FA-G)	5	5.9200	.2683	5	5.8600	.2510	A	10	5.8900	.2470	4.19
LAB- 8 (AA)	5	3.2060	.0573	5	3.1900	.0548	A	10	3.1980	.0535	1.67
LAB- 9 (AA)	5	5.3000	.0707	5	5.3800	.0837	A	10	5.3400	.0843	1.58
LAB-12 (FA-AA)	5	3.9580	.0303	5	4.0220	.0311	REJECT	10	3.9900	.0445	1.11
LAB-14 (AA)	5	6.0600	.0548	5	6.0200	.0447	A	10	6.0400	.0516	.85
LAB-15 (FA-AA)	5	4.6400	.5177	5	4.6800	.1789	A	10	4.6600	.3658	7.85
LAB-16 (AA)	5	3.8640	.1203	5	3.8100	.0548	A	10	3.8370	.0926	2.41
LAB-19 (FA-AA)	5	4.2620	.0396	5	4.3320	.0630	A	10	4.2970	.0618	1.44
LAB-23 (AA)	5	6.2400	.2302	5	6.3800	.2168	A	10	6.3100	.2234	3.54
LAB-24 (FA-AA)	5	4.6380	.3152	5	4.9800	.4699	A	10	4.8090	.4181	8.69
LAB-28 (AA)	5	4.5200	.1643	5	4.1000	.1225	REJECT	10	4.3100	.2601	6.04
LAB-30 (AA)	5	3.4000	.1000	5	3.2400	.1673	A	10	3.3200	.1549	4.67
LAB-31 (AA)	5	5.5400	.2608	5	5.4400	.3578	A	10	5.4900	.2998	5.46
LAB-33 (AA)	5	4.1400	.0894	5	4.0800	.1304	A	10	4.1100	.1101	2.68
LAB-33 (ES)	5	4.0000	.3536	5	4.2000	.2739	A	10	4.1000	.3162	7.71
LAB-36 (ES)	5	5.2000	.4183	5	4.9800	.6760	A	10	5.0900	.5425	10.66

Variance between sets, between bottles and within bottles = 8.42×10^{-1} , 1.52×10^{-3} and 6.19×10^{-2} , respectively.

Table 6 (cont'd)

		Pt ($\mu\text{g/g}$)										
		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 5	(FA-COL)	5	.4960	.0219	5	.5120	.0277	A	10	.5040	.0250	4.97
LAB- 6	(FA-AA)	4	.3025	.0222	5	.3040	.0182	A	9	.3033	.0187	6.17
LAB-12	(FA-ES)	5	.3550	.0378	5	.3660	.0329	A	10	.3610	.0338	9.37
LAB-15	(FA-ES)	5	.4840	.0594	5	.3920	.0045	REJECT	10	.4380	.0627	14.31
LAB-19	(FA-ES)	5	.4120	.0084	5	.4100	.0612	A	10	.4110	.0412	10.03
LAB-23	(FA-AA)	5	.5334	.0154	5	.5350	.0087	A	10	.5342	.0118	2.21
LAB-24	(FA-ES)	5	.3560	.0219	5	.4120	.0460	REJECT	10	.3840	.0450	11.72
LAB-26	(NAA)	3	.4397	.0540	3	.3873	.0348	A	6	.4135	.0497	12.02
LAB-33	(FA-ES)	5	.3500	.0354	5	.3420	.0402	A	10	.3460	.0360	10.39

Variance between sets, between bottles and within bottles = 4.66×10^{-3} , 6.17×10^{-4} and 1.21×10^{-3} , respectively.

		Pd ($\mu\text{g/g}$)										
		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V. (%)
LAB- 5	(FA-COL)	5	.3500	.0141	5	.3580	.0110	A	10	.3540	.0126	3.57
LAB- 6	(FA-AA)	5	.3840	.0195	5	.3440	.0134	REJECT	10	.3640	.0263	7.23
LAB-12	(FA-ES)	5	.3580	.0130	5	.3560	.0114	A	10	.3570	.0116	3.25
LAB-15	(FA-ES)	5	.4380	.0205	5	.4300	.0283	A	10	.4340	.0237	5.45
LAB-18	(FA-AA)	2	.3625	.0205	2	.3770	0.0000	A	4	.3698	.0145	3.92
LAB-19	(FA-ES)	5	.3560	.0114	5	.3620	.0179	A	10	.3590	.0145	4.04
LAB-23	(FA-AA)	5	.3274	.0208	5	.3280	.0217	A	10	.3277	.0200	6.12
LAB-24	(FA-AA)	5	.3854	.0189	5	.3966	.0200	A	10	.3910	.0193	4.93
LAB-26	(NAA)	3	.3723	.0131	3	.3873	.0170	A	6	.3798	.0159	4.18
LAB-33	(FA-ES)	5	.1660	.0134	5	.1680	.0179	A	10	.1670	.0149	8.95

Variance between sets, between bottles and within bottles = 6.05×10^{-3} , 5.60×10^{-5} and 3.13×10^{-4} , respectively.

Table 6 (cont'd)

		Ni (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)	THERE ARE MORE THAN 2 BOTTLES										
LAB- 1	(COLOR)	5	1.2320	.0115	5	1.2428	.0088	A	10	1.2374	.0112	.91
LAB- 6	(AA)	5	1.2320	.0045	5	1.2260	.0055	A	10	1.2290	.0057	.46
LAB- 8	(AA)	5	1.2062	.0053	5	1.2058	.0019	A	10	1.2060	.0037	.31
LAB- 9	(AA)	5	1.2120	.0192	5	1.2320	.0084	A	10	1.2220	.0175	1.43
LAB-12	(AA)	5	1.2080	.0027	5	1.2060	.0022	A	10	1.2070	.0026	.21
LAB-12	(XRF)	5	1.2420	.0121	5	1.2362	.0136	A	10	1.2391	.0125	1.01
LAB-14	(AA)	5	1.2300	.0000	5	1.2300	.0000	A	10	1.2300	.0000	.00
LAB-14	(GRAV)	5	1.2340	.0055	5	1.2260	.0055	REJECT	10	1.2300	.0067	.54
LAB-15	(TITR)	5	1.2542	.0018	5	1.2568	.0018	A	10	1.2555	.0022	.17
LAB-16	(AA)	5	1.1920	.0045	5	1.2040	.0055	REJECT	10	1.1980	.0079	.66
LAB-17	(AA)	5	.9580	.0045	5	.9580	.0045	A	10	.9580	.0042	.44
LAB-18	(AA)	5	1.2162	.0156	5	1.2126	.0092	A	10	1.2144	.0122	1.00
LAB-19	(AA)	5	1.2384	.0061	5	1.2368	.0052	A	10	1.2376	.0054	.44
LAB-20	(AA)	5	1.2376	.0021	5	1.2324	.0018	REJECT	10	1.2350	.0033	.27
LAB-20	(GRAV)	5	1.2422	.0055	5	1.2384	.0067	A	10	1.2403	.0061	.50
LAB-21	(AA)	5	1.2420	.0107	5	1.2300	.0122	A	10	1.2360	.0125	1.01
LAB-23	(AA)	5	1.2460	.0032	5	1.2482	.0027	A	10	1.2471	.0030	.24
LAB-24	(AA)	5	1.2072	.0103	5	1.2228	.0099	REJECT	10	1.2150	.0126	1.03
LAB-28	(AA)	5	1.2560	.0089	5	1.2400	.0187	A	10	1.2480	.0162	1.30
LAB-28	(AA)	6	1.2200	.0063	6	1.2250	.0105	A	12	1.2225	.0087	.71
LAB-30	(GRAV)	5	1.2500	.0071	5	1.2580	.0045	A	10	1.2540	.0070	.56
LAB-31	(AA)	5	1.2268	.0047	5	1.2256	.0037	A	10	1.2262	.0040	.33
LAB-33	(AA)	5	1.1360	.0590	5	1.0920	.0084	A	10	1.1140	.0460	4.13
LAB-34	(XRF)	5	1.0508	.0255	5	1.0482	.0286	A	10	1.0495	.0256	2.44

Variance between sets, between bottles and within bottles = 4.70×10^{-3} , 3.95×10^{-5} and 1.59×10^{-4} , respectively.

Table 6 - Laboratory means, coefficients of variation and summary of t-test on
between bottle results for certified constituents

		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	(AA)	5	.9634	.0056	5	.9678	.0079	A	10	.9656	.0069	.71
LAB- 1	(COLOR)	5	.9732	.0157	5	.9810	.0162	A	10	.9771	.0156	1.60
LAB- 6	(AA)	5	.9800	.0141	5	.9720	.0110	A	10	.9760	.0126	1.30
LAB- 8	(AA)	5	.9114	.0049	5	.9118	.0051	A	10	.9116	.0047	.52
LAB- 9	(AA)	5	.9714	.0087	5	.9798	.0080	A	10	.9756	.0091	.93
LAB- 9	(ELECTR)	5	.9790	.0071	5	.9760	.0084	A	10	.9775	.0075	.77
LAB-12	(AA)	5	.9520	.0027	5	.9520	.0027	A	10	.9520	.0026	.27
LAB-12	(XRF)	5	.9658	.0078	5	.9648	.0158	A	10	.9653	.0118	1.22
LAB-14	(AA)	5	.9580	.0045	5	.9580	.0045	A	10	.9580	.0042	.44
LAB-14	(ELECTR)	5	.9700	.0071	5	.9740	.0055	A	10	.9720	.0063	.65
LAB-14	(ELECTR)	5	.9716	.0017	5	.9708	.0011	A	10	.9712	.0014	.14
LAB-15	(AA)	5	.9396	.0072	5	.9384	.0048	A	10	.9390	.0058	.62
LAB-16	(AA)	5	.9670	.0027	5	.9710	.0042	A	10	.9690	.0039	.41
LAB-17	(AA)	5	.9960	.0055	5	.9880	.0045	REJECT	10	.9920	.0063	.64
LAB-18	(AA)	5	.9560	.0114	5	.9540	.0055	A	10	.9550	.0085	.89
LAB-19	(AA)	5	.9800	.0040	5	.9808	.0018	A	10	.9804	.0030	.30
LAB-20	(AA)	5	.9576	.0027	5	.9564	.0025	A	10	.9570	.0025	.27
LAB-21	(AA)	5	.9690	.0091	5	.9550	.0033	REJECT	10	.9620	.0098	1.02
LAB-23	(AA)	5	.9550	.0010	5	.9556	.0030	A	10	.9553	.0021	.22
LAB-24	(AA)	5	.9650	.0082	5	.9698	.0108	A	10	.9674	.0094	.97
LAB-28	(AA)	5	.9720	.0045	5	.9720	.0084	A	10	.9720	.0063	.65
LAB-28	(AA)	5	.9600	.0063	6	.9550	.0122	A	12	.9575	.0097	1.01
LAB-30	(TITR)	5	.9200	.0173	5	.9160	.0313	A	10	.9180	.0239	2.61
LAB-31	(AA)	5	.9828	.0043	5	.9768	.0032	REJECT	10	.9798	.0048	.49
LAB-33	(AA)	5	.9750	.0152	5	.9720	.0130	A	10	.9740	.0135	1.39
LAB-34	(XRF)	5	.9638	.0104	5	.9706	.0140	A	10	.9672	.0122	1.26

Variance between sets, between bottles and within bottles = 3.20×10^{-4} , $0.$ and 8.83×10^{-5} , respectively.

Table 7 - Analytical results for reference ore SU-1a

REFERENCE ORE SU-1A										
NICKEL (WT %)										
LAB- 1 (AA)	1.26	1.26	1.26	1.25	1.25	1.25	1.26	1.25	1.25	1.25
	1.24	1.25	1.24	1.25	1.26	1.26	1.25	1.26	1.26	1.25
	1.24	1.26	1.25	1.27	1.26	1.24	1.25	1.24	1.25	1.26
LAB- 6 (AA)	1.24	1.23	1.23	1.23	1.23	1.22	1.22	1.23	1.23	1.23
LAB- 8 (AA)	1.203	1.200	1.207	1.214	1.207	1.203	1.206	1.208	1.207	1.205
LAB- 9 (AA)	1.19	1.22	1.21	1.20	1.24	1.24	1.22	1.24	1.23	1.23
LAB-12 (AA)	1.205	1.210	1.210	1.205	1.210	1.205	1.205	1.205	1.210	1.205
LAB-14 (AA)	1.23	1.23	1.23	1.23	1.23	1.23	1.23	1.23	1.23	1.23
LAB-16 (AA)	1.20	1.19	1.19	1.19	1.19	1.21	1.20	1.20	1.21	1.20
LAB-17 (AA)	0.96	0.96	0.96	0.96	0.95	0.96	0.96	0.96	0.95	0.96
LAB-18 (AA)	1.210	1.210	1.220	1.200	1.241	1.220	1.210	1.210	1.200	1.223
LAB-19 (AA)	1.244	1.240	1.244	1.232	1.232	1.240	1.244	1.236	1.232	1.232
LAB-20 (AA)	1.236	1.239	1.240	1.235	1.238	1.233	1.231	1.230	1.234	1.234
LAB-21 (AA)	1.252	1.231	1.250	1.230	1.247	1.239	1.221	1.237	1.240	1.213
LAB-23 (AA)	1.244	1.248	1.246	1.242	1.25	1.248	1.25	1.251	1.244	1.248
LAB-24 (AA)	1.199	1.211	1.223	1.205	1.198	1.224	1.221	1.216	1.214	1.239
LAB-28 (AA)	1.26	1.24	1.26	1.26	1.26	1.26	1.25	1.24	1.24	1.21
LAB-28 (AA)	1.23	1.22	1.22	1.21	1.22	1.22	1.23	1.23	1.24	1.22
	1.21	1.22								
LAB-31 (AA)	1.234	1.225	1.223	1.229	1.223	1.232	1.225	1.223	1.223	1.225
LAB-33 (AA)	1.20	1.10	1.20	1.08	1.10	1.09	1.08	1.09	1.10	1.10
LAB-15 (TITR)	1.252	1.254	1.254	1.257	1.254	1.257	1.254	1.257	1.259	1.257
LAB- 1 (COLOR)	1.225	1.216	1.243	1.242	1.234	1.250	1.254	1.238	1.239	1.233
LAB-14 (GRAV)	1.23	1.24	1.23	1.23	1.24	1.23	1.23	1.22	1.23	1.22
LAB-20 (GRAV)	1.233	1.246	1.245	1.241	1.246	1.228	1.245	1.243	1.240	1.236
LAB-30 (GRAV)	1.25	1.24	1.25	1.26	1.25	1.25	1.26	1.26	1.26	1.26
LAB-12 (XRF)	1.262	1.239	1.243	1.230	1.236	1.229	1.224	1.232	1.259	1.237
LAB-34 (XRF)	1.048	1.072	1.066	1.060	1.008	1.007	1.060	1.063	1.032	1.079

Table 7 (cont'd)

COPPER (WT %)										
LAB- 1 (AA)	.964	.960	.960	.960	.973	.969	.963	.957	.973	.977
LAB- 6 (AA)	1.00	.98	.98	.98	.96	.96	.96	.98	.98	.98
LAB- 8 (AA)	.914	.916	.904	.909	.914	.914	.907	.907	.919	.912
LAB- 9 (AA)	.966	.979	.979	.959	.974	.979	.967	.989	.982	.982
LAB-12 (AA)	.950	.950	.955	.955	.950	.955	.955	.950	.950	.950
LAB-14 (AA)	.96	.95	.96	.96	.96	.95	.96	.96	.96	.96
LAB-15 (AA)	.944	.940	.947	.939	.928	.936	.945	.942	.935	.934
LAB-16 (AA)	.965	.970	.965	.970	.965	.975	.970	.965	.975	.970
LAB-17 (AA)	.99	1.00	1.00	.99	1.00	.98	.99	.99	.99	.99
LAB-18 (AA)	.940	.960	.960	.950	.970	.960	.950	.950	.950	.960
LAB-19 (AA)	.984	.976	.976	.980	.984	.980	.984	.980	.980	.980
LAB-20 (AA)	.957	.955	.958	.962	.956	.958	.957	.957	.952	.958
LAB-21 (AA)	.980	.960	.976	.960	.969	.956	.960	.952	.952	.955
LAB-23 (AA)	.955	.954	.956	.956	.954	.96	.956	.954	.956	.952
LAB-24 (AA)	.962	.952	.972	.969	.970	.960	.958	.974	.973	.984
LAB-28 (AA)	.97	.97	.97	.98	.97	.98	.98	.96	.97	.97
LAB-28 (AA)	.96	.95	.96	.97	.96	.96	.97	.96	.96	.94
	.96	.94								
LAB-31 (AA)	.982	.983	.990	.979	.980	.975	.979	.976	.981	.973
LAB-33 (AA)	.96	.99	.98	.99	.96	.96	.98	.99	.96	.97
LAB-30 (TITR)	.92	.93	.93	.89	.93	.93	.93	.86	.93	.93
LAB- 1 (COLOR)	.960	.996	.957	.974	.979	.977	.996	1.000	.966	.966
LAB-12 (XRF)	.962	.961	.959	.978	.969	.954	.961	.949	.989	.971
LAB-34 (XRF)	.979	.954	.965	.967	.954	.972	.984	.974	.947	.976
LAB- 9 (ELECTR)	.987	.983	.968	.978	.979	.979	.979	.967	.987	.968
LAB-14 (ELECTR)	.96	.97	.97	.97	.98	.98	.97	.97	.98	.97
LAB-14 (ELECTR)	.970	.974	.972	.972	.970	.970	.970	.972	.970	.972

Table 7 (cont'd)

REFERENCE ORE SU-1A

COBALT (WT %)

LAB- 1 (AA)	.0380	.0377	.0380	.0380	.0386	.0386	.0380	.0383	.0383	.0386
LAB- 6 (AA)	.042	.042	.042	.042	.042	.042	.042	.042	.042	.042
LAB- 8 (AA)	.0242	.0243	.0244	.0245	.0248	.0243	.0244	.0244	.0247	.0247
LAB- 9 (AA)	.042	.042	.040	.040	.042	.042	.042	.042	.042	.042
LAB-12 (AA)	.0395	.0400	.0395	.0395	.0400	.0400	.0400	.0395	.0395	.0400
LAB-14 (AA)	.039	.038	.038	.040	.038	.038	.038	.040	.038	.038
LAB-15 (AA)	.040	.040	.040	.040	.040	.041	.042	.042	.041	.041
LAB-16 (AA)	.0388	.0392	.0384	.0392	.0388	.0392	.0392	.0388	.0392	.0388
LAB-17 (AA)	.042	.042	.042	.042	.042	.042	.042	.042	.042	.042
LAB-18 (AA)	.0430	.0440	.0420	.0430	.0470	.0410	.0420	.0410	.0420	.0460
LAB-19 (AA)	.0404	.0404	.0404	.0390	.0390	.0404	.0400	.0402	.0396	.0396
LAB-20 (AA)	.040	.040	.040	.040	.040	.040	.040	.040	.041	.040
LAB-21 (AA)	.0406	.0406	.0402	.0402	.0406	.0404	.0400	.0401	.0402	.0403
LAB-23 (AA)	.047	.047	.048	.047	.047	.048	.047	.048	.047	.048
LAB-24 (AA)	.0398	.0397	.0399	.0408	.0390	.0405	.0390	.0401	.0398	.0385
LAB-28 (AA)	.0425	.0414	.0425	.0414	.0421	.0427	.0425	.0419	.0418	.0404
LAB-30 (AA)	.032	.032	.032	.032	.032	.033	.033	.033	.034	.032
LAB-31 (AA)	.0397	.0384	.0390	.0393	.0395	.0396	.0395	.0401	.0394	.0400
LAB-33 (AA)	.043	.040	.040	.043	.040	.040	.039	.041	.040	.040
LAB- 1 (COLOR)	.0380	.0393	.0397	.0380	.0385	.0366	.0380	.0380	.0403	.0385
LAB-12 (XRF)	.035	.032	.043	.045	.048	.042	.034	.041	.047	.044
LAB-34 (XRF)	.046	.048	.046	.049	.044	.046	.050	.048	.045	.046

SILVER (UG/G)

LAB- 8 (AA)	3.25	3.21	3.21	3.25	3.11	3.25	3.25	3.15	3.15	3.15
LAB- 9 (AA)	5.3	5.2	5.3	5.3	5.4	5.4	5.5	5.4	5.3	5.3
LAB-14 (AA)	6.1	6.0	6.0	6.1	6.1	6.0	6.0	6.0	6.1	6.0
LAB-16 (AA)	3.75	3.85	3.95	4.02	3.75	3.85	3.75	3.85	3.85	3.75
LAB-23 (AA)	6.4	6.0	6.3	6.5	6.0	6.2	6.5	6.6	6.5	6.1
LAB-28 (AA)	4.4	4.4	4.5	4.5	4.8	4.0	4.3	4.0	4.1	4.1
LAB-30 (AA)	3.5	3.3	3.5	3.4	3.3	3.2	3.0	3.4	3.4	3.2
LAB-31 (AA)	5.5	5.4	5.8	5.2	5.8	5.3	5.3	5.7	5.9	5.0
LAB-33 (AA)	4.2	4.2	4.2	4.0	4.1	3.9	4.0	4.1	4.2	4.2
LAB- 6 (FA-G)	5.8	6.1	5.5	6.1	6.1	6.1	5.8	5.5	5.8	6.1
LAB- 5 (FA-AA)	4.5	4.2	4.1	4.5	4.2	4.1	4.5	4.3	4.1	4.1
LAB-12 (FA-AA)	3.94	3.93	3.98	3.94	4.00	3.99	4.01	4.06	4.00	4.05
LAB-15 (FA-AA)	5.1	4.2	4.3	5.3	4.3	4.5	4.5	4.7	4.9	4.8
LAB-19 (FA-AA)	4.20	4.26	4.27	4.27	4.31	4.37	4.28	4.32	4.42	4.27
LAB-24 (FA-AA)	4.45	4.69	4.27	4.67	5.11	4.58	4.41	5.07	5.38	5.46
LAB-33 (ES)	4.0	3.5	4.0	4.0	4.5	4.5	4.0	4.0	4.5	4.0
LAB-36 (ES)	5.6	4.7	5.4	4.8	5.5	5.6	4.7	4.3	5.8	4.5

Table 7 (cont'd)

PLATINUM (UG/G)										
LAB-26 (NAA)	.410	.407	.502	.373	.427	.362				
LAB- 6 (FA-AA)	.29	.33	.28	.31	.29	.32	.32	.28	.31	
LAB-23 (FA-AA)	.53	.56	.527	.52	.53	.535	.53	.55	.53	.53
LAB-12 (FA-ES)	.36	.30	.34	.39	.39	.34	.33	.40	.36	.40
LAB-15 (FA-ES)	.44	.56	.53	.47	.42	.39	.40	.39	.39	.39
LAB-19 (FA-ES)	.41	.42	.40	.41	.42	.39	.48	.36	.47	.35
LAB-24 (FA-ES)	.34	.34	.38	.34	.38	.38	.48	.38	.38	.44
LAB-33 (FA-ES)	.35	.35	.30	.35	.40	.38	.38	.30	.35	.30
LAB- 5 (FA-COL)	.52	.48	.48	.48	.52	.55	.48	.53	.50	.50

PALLADIUM (UG/G)										
LAB-26 (NAA)	.368	.362	.387	.378	.407	.377				
LAB- 6 (FA-AA)	.39	.35	.40	.39	.39	.33	.35	.33	.35	.36
LAB-18 (FA-AA)	.348	.377	.377	.377						
LAB-23 (FA-AA)	.317	.35	.31	.31	.35	.33	.30	.36	.32	.33
LAB-24 (FA-AA)	.364	.368	.390	.397	.408	.397	.364	.399	.405	.418
LAB-12 (FA-ES)	.34	.36	.37	.37	.35	.34	.37	.35	.36	.36
LAB-15 (FA-ES)	.44	.42	.47	.42	.44	.45	.44	.38	.44	.44
LAB-19 (FA-ES)	.34	.37	.35	.36	.36	.39	.36	.36	.34	.36
LAB-33 (FA-ES)	.16	.18	.18	.15	.16	.16	.18	.18	.14	.18
LAB- 5 (FA-COL)	.36	.37	.34	.34	.34	.34	.37	.36	.36	.36

Table 8 - Analytical results for gold and secondary precious metals

Element	Lab No.	Method	No. of Results	($\mu\text{g/g}$)
Au	15	Fire assay - emission spectrography	10	0.15
	19	" "	10	0.15
	26	Neutron activation analysis	6	0.17
Ir	26	" "	6	0.025
Os	26	" "	6	0.0109
Rh	15	Fire assay - emission spectrography	10	0.09
	26	Neutron activation analysis	6	0.071
Ru	26	" "	6	0.056

Table 9 - Analytical results by emission spectrography for other elements

Element	Overall mean ($\mu\text{g/g}$)*	Element	Overall mean ($\mu\text{g/g}$)*
Ba	1040	V	157
Ge	1.1	Y	34
Pb	77	Zr	92
Sr	713		

*Mean of 10 determinations by Laboratory 33.

DISCUSSION OF ANALYTICAL RESULTS

Table 5 is a summary of a methodological classification of accepted analytical results where there is a clear-cut distinction between types of methods in decomposition, separations and determinative steps. The results for nickel, copper and cobalt obtained by atomic absorption pertain to a single solution prepared from each sub-sample of SU-1a.

RECOMMENDED VALUE FOR SILVER

The overall mean of 17 sets of silver results was $4.7 \mu\text{g/g}$ with a 95% confidence interval of $\pm 0.5 \mu\text{g/g}$. An examination of Fig. 1 however, illustrates the relatively wide range in silver results which can most probably be attributed to the low silver content of SU-1a. Accordingly, it was decided that the overall mean was not the most

suitable estimate of the silver content and the following procedure was employed to arrive at the recommended value.

The overall mean and standard deviation of the five accepted sets of results obtained by a fire-assay concentration step were calculated. The results from laboratory 6 were rejected because it was considered that fire assay with gravimetric finish was unsuitable at the low silver content of SU-1a. Moreover, no correction was made for gold, iridium and rhodium of which a total of approximately $0.3 \mu\text{g/g}$ was present. The 10 sets of results, of which the individual mean lay with 2σ of the overall mean of the 5 fire-assay sets, were included in the calculation of a new overall mean and recommended value of $4.3 \mu\text{g/g}$ with 95% confidence intervals of $\pm 0.3 \mu\text{g/g}$ for silver in SU-1a.

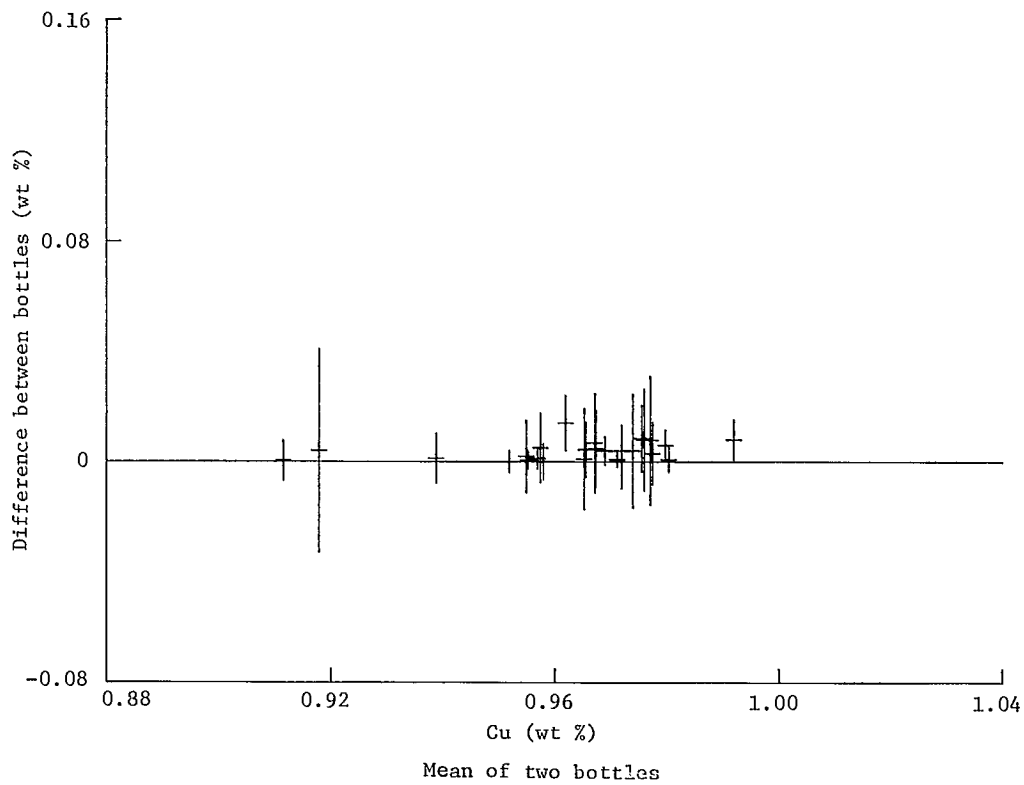
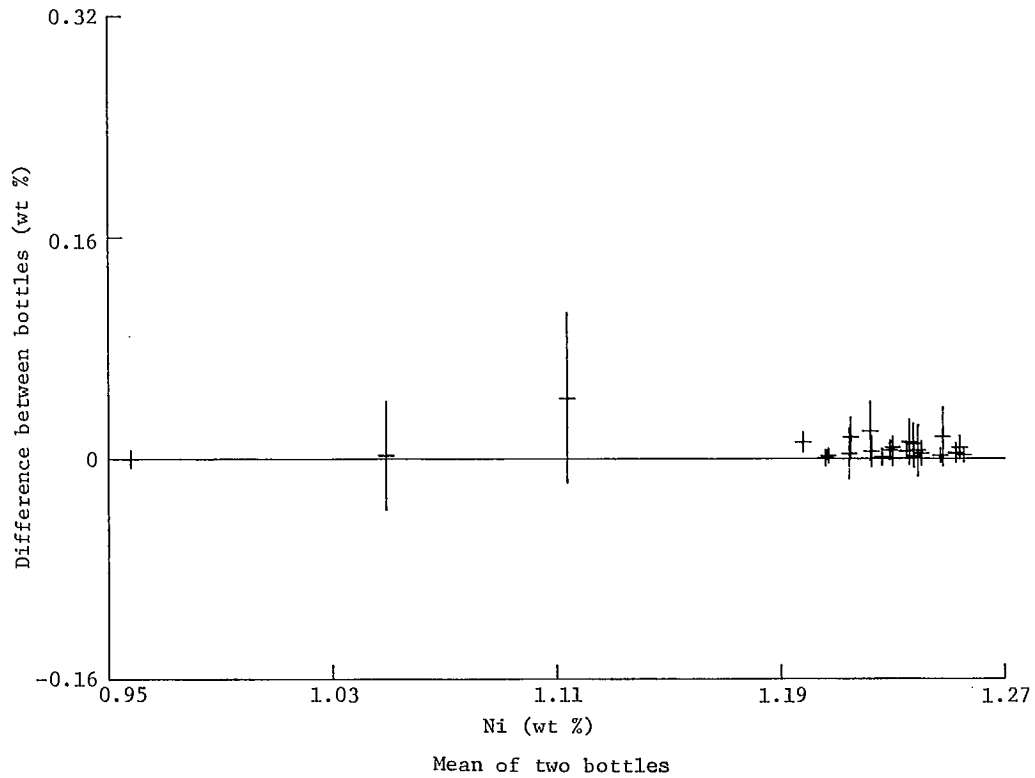


Fig. 1 - Degree of homogeneity of SU-1a. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.

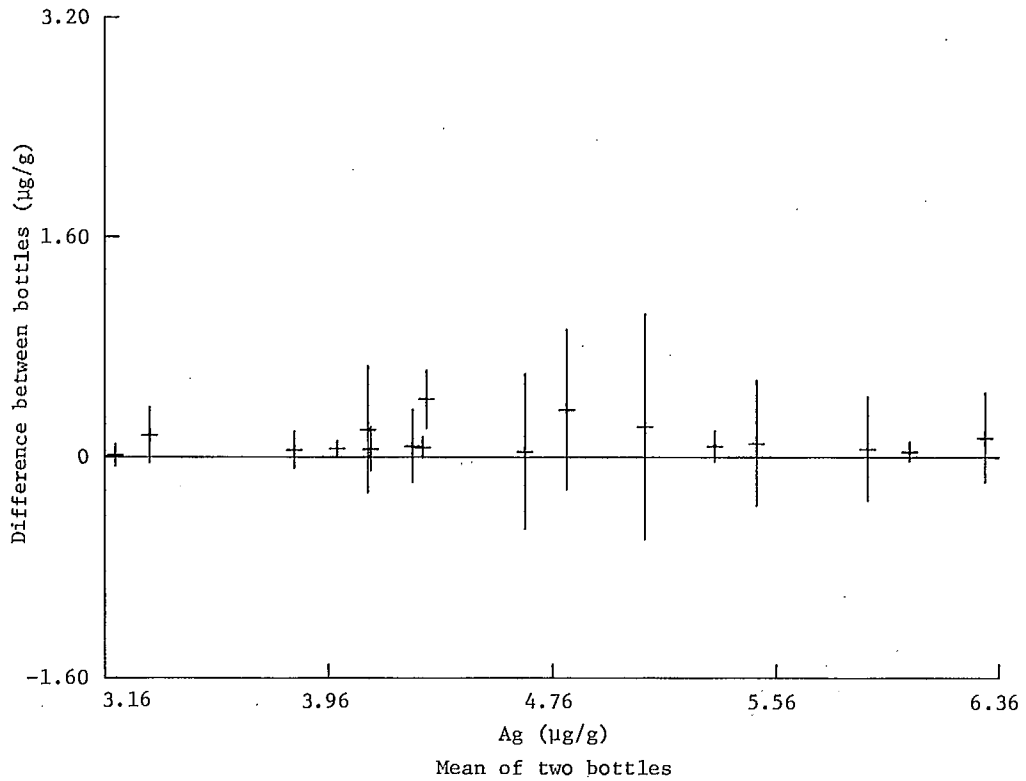
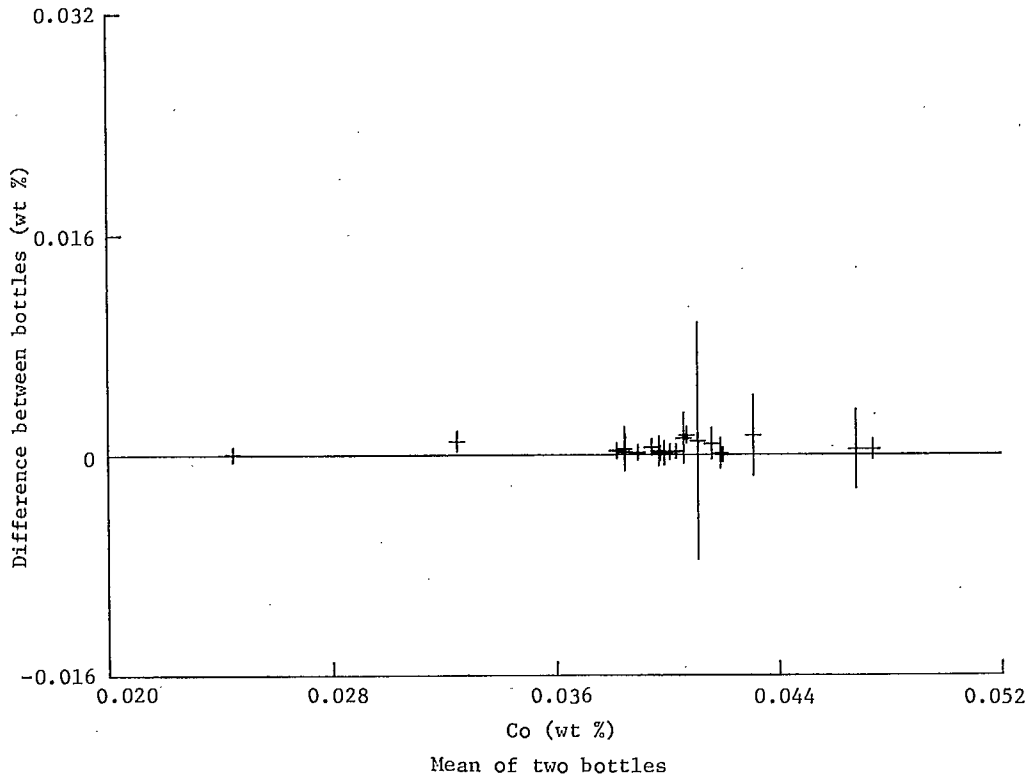


Fig. 1 - Degree of homogeneity of SU-1a. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.

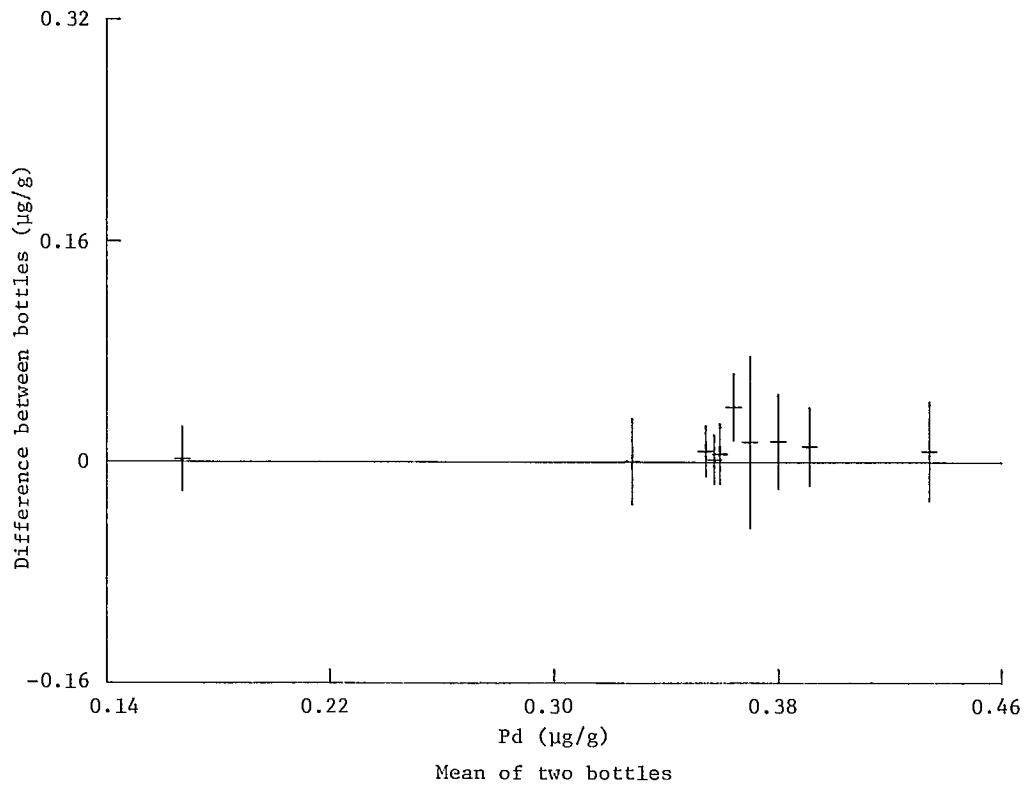
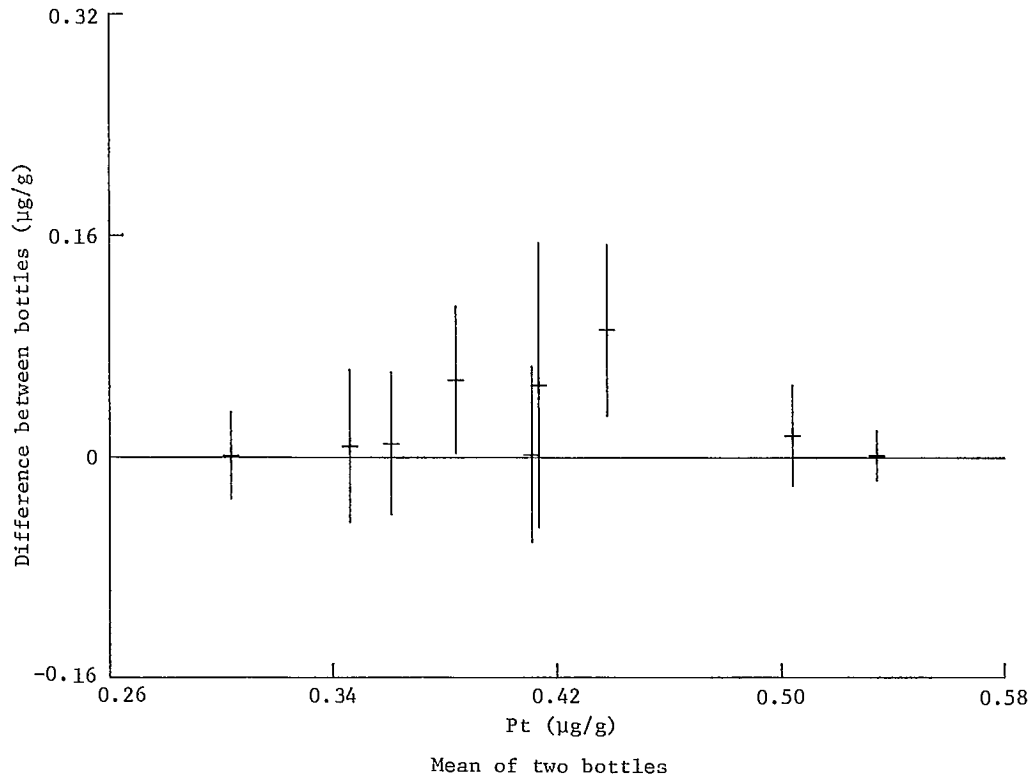
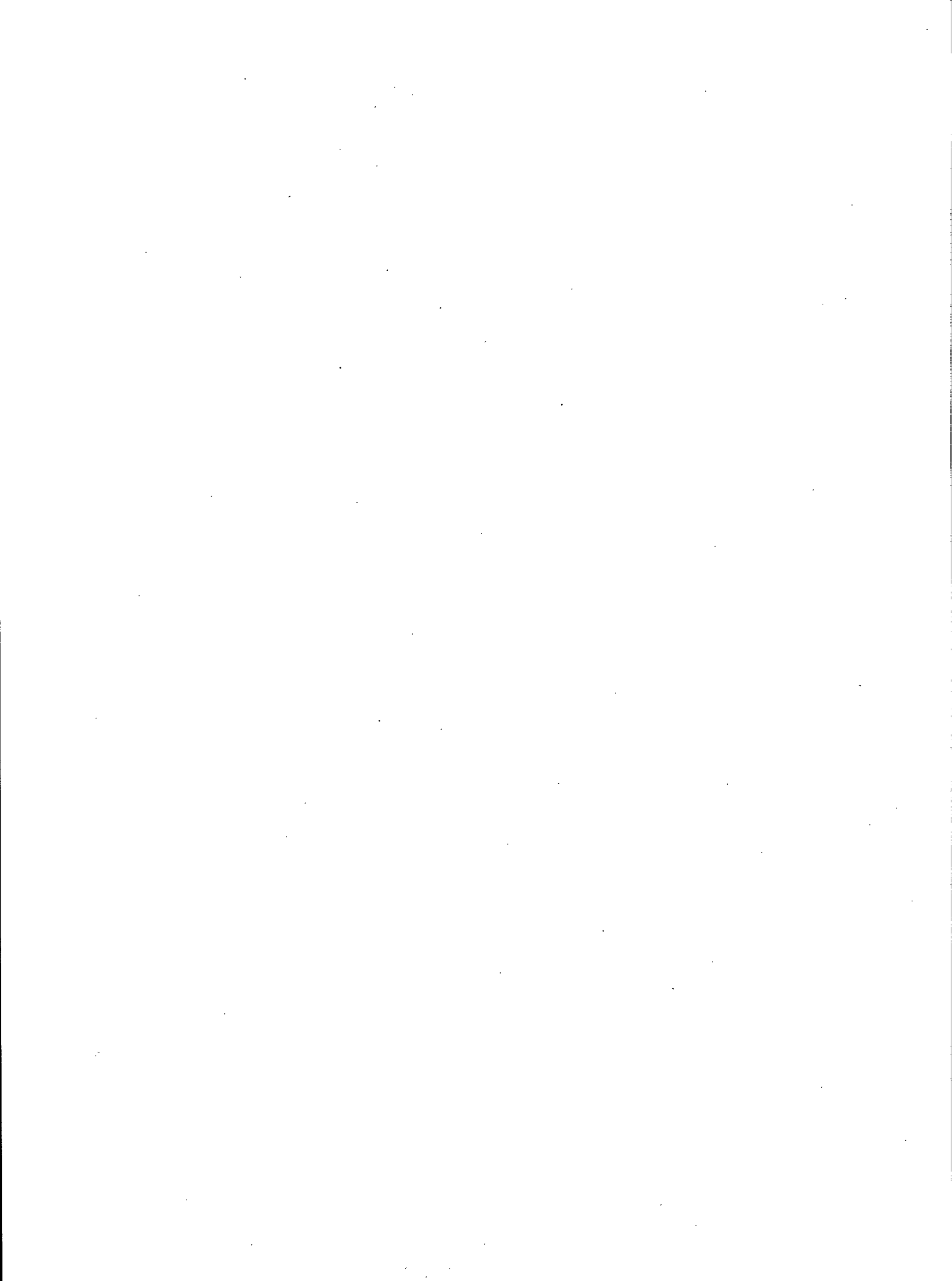


Fig. 1 - Degree of homogeneity of SU-1a. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory.

REFERENCES

1. Steger, H.F. "Certified reference materials"; CANMET Report 80-6E; CANMET, Energy, Mines and Resources Canada; 1980.
2. Faye, G.H., Bowman, W.S. and Sutarno, R. "Nickel-copper-cobalt ores SU-1 and UM-1: Their characterization and preparation for use as standard reference materials"; Mines Branch [since renamed CANMET] Technical Bulletin TB 177; CANMET, Energy, Mines and Resources Canada; 1973.
3. Brownlee, K.A. "Statistical theory and methodology in science and engineering"; John Wiley and Sons, Inc., New York; 1960.

APPENDIX A



PARTICIPATING LABORATORIES

Bondar-Clegg and Company Ltd., Ottawa, Ontario.	Geological Survey of Norway, Trondheim, Norway.
Bondar-Clegg and Company Ltd., Whitehorse, Yukon.	Geological Survey of West Malaysia, Ipoh, Perab, Malaysia.
British Columbia Department of Energy, Mines and Petroleum Resources, Victoria, British Columbia.	Hudson Bay Mining and Smelting Company Ltd., Flin Flon, Manitoba.
CANMET, Energy, Mines and Resources Canada, Mineral Sciences Laboratories, Ottawa, Ontario (three independent analysts)	Inco Ltd., Analytical Services, Process Technology, Copper Cliff, Ontario.
Chemex Labs. Ltd., North Vancouver, British Columbia.	Inco Ltd., J. Roy Gordon Research Laboratory, Sheridan Park, Ontario.
Department of Geology, University of Toronto, Toronto, Ontario.	Lakefield Research of Canada Ltd., Lakefield, Ontario.
Falconbridge Nickel Mines Ltd., Metallurgical Laboratories, Thornhill, Ontario.	National Institute for Metallurgy, Randburg, South Africa.
Falconbridge Nickel Mines Ltd., Sudbury Division, Falconbridge, Ontario.	Noranda Mines Ltd., Noranda, Quebec.
Geological Survey Department, Lusaka, Zambia.	Geoscience Laboratories, Ontario Ministry of Natural Resources, Mineral Research Branch, Toronto, Ontario.
Geological Survey of India, Central Chemical Laboratory, Calcutta, India.	U.S. Bureau of Mines, Reno Metallurgy Research Center, Reno, Nevada.

