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DL-1a: A CERTIFIED URANIUM-THORIUM REFERENCE ORE

H.F. STEGER AND W.S. BOWMAN

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DL-1a: A CERTIFIED URANIUM-THORIUM REFERENCE ORE

by

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

SYNOPSIS

A 338-kg sample of a uranium-thorium ore, DL-1a, from Elliot Lake, Ontario, was prepared as a compositional reference material to replace the similar certified ore, DL-1, of which the stock had been exhausted. DL-1a was ground to minus 74 µm, blended in one lot, tested for homogeneity by optical fluorimetric and chemical methods and bottled in 200-g units.

In a "free choice" analytical program, 20 laboratories contributed results for one or both of uranium and thorium in each of two bottles of DL-1a. A statistical analysis of the data gave a recommended value of 0.0116±0.0003% for U and 0.0076±0.0004% for thorium.

Information is also presented on the state of secular equilibrium of DL-1a and on its Ra-226 content. Values of the iron, sulphur and lead content of DL-1a are also presented for information purposes.

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

DL-1a: MINERAI DE REFERENCE CERTIFIE D'URANIUM-THORIUM

par

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

SYNOPSIS

Un échantillon de 338 kg de minerai d'uranium-thorium, DL-1a, provenant d'Elliot lake (Ontario), a été préparé comme matériau de référence de composition pour remplacer le minerai certifié analogue, DL-1, dont l'inventaire était épuisé. Le DL-1a a été broyé à une granulométrie de moins 74 µm, mélangé en lot de minerai, soumis à des essais d'homogénéité par la méthode de fluorimétrie optique et autres méthodes chimiques et embouteillé en unités de 200 g.

En vertu d'un programme analytique de "libre choix", 20 laboratoires ont soumis les résultats sur chacun des deux flacons de DL-1a pour un ou les deux éléments, uranium et thorium. L'analyse statistique des données a donné des valeurs recommandées de 0,0116±0,0003% pour l'uranium et 0,0076±0,0004% pour le thorium.

L'information est aussi présentée sur l'état d'équilibre séculaire du DL-1a et sa teneur de Ra-226. Les valeurs pour le fer, le soufre et le plomb ont aussi été déterminées à titre de renseignements.

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Note: D'autres membres des Laboratoires des sciences minérales ont aussi contribué à l'homologation du DL-1a.

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INTRODUCTION

The preparation, characterization and certification of the low-grade uranium ore DL-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 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).

DL-1a was intended to replace DL-1 the supply of which was exhausted (2). The latter was part of a popular suite of seven uranium-thorium reference samples identified as DH-1, DL-1, BL-1 to BL-4 (2) and BL-5 (3) and had been selected by the Analytical Sub-Committee of the Canadian Uranium Producers Metallurgical Committee for establishing the "state-of-the-art" of radium determination in the uranium industry.

An interlaboratory program was conducted to obtain results for uranium and thorium from twenty commercial, industrial and government laboratories using analytical methods of their own choice. The results should therefore be indicative of the current "state-of-the-art" of uranium and thorium analysis.

NATURE AND PREPARATION

The raw material for DL-1a was donated to CCRMP in July 1978 by Denison Mines Ltd. and was a waste-rock typical of the Denison property in Elliot Lake, Ontario. It was a pale yellow arkose sandstone containing uraninite and brannerite and possibly traces of monazite and uranothorite.

DL-1a was dry-ground in November 1978 to pass a 74-µm screen. The powdered ore weighing approximately 338 kg was tumbled in a 570-L conical blender for 9 h and then sampled systematically for analysis for uranium by an optical fluorimetric method. It was found sufficiently homogeneous to qualify as a reference material and was bottled in 200-g units. The approximate chemical composition and particle size analysis are given in Tables 1 and 2.

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 uranium and thorium on each of two bottles of DL-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

Table 1 - Approximate chemical composition of DL-1a

Element	wt %*
Si	39.95
Al	5.25
Fe	0.90
S	0.42
Ca	0.29
K	0.23
Mg	0.19
Na	0.09
Ti	0.09
LOI (900°C)	1.43
H_2O (105°C)	0.18

*Mean of a minimum of three determinations.

Table 2 - Particle size analysis (wet screen)

Size of fraction (µm)	wt %*
-104 + 74	1
- 74 + 55	15
- 55 + 46	4
- 46 + 37	10
- 37	70

*Mean of duplicate determinations.

or contributed results for one element only. Where a laboratory submitted results by more than one method, each set was considered statistically independent.

Some laboratories also reported results for one or more of iron, sulphur and lead contents and radium-226 activity. These are reported for the information of users of DL-1a.

The recommended values for uranium and thorium are presented in Table 3. Methodological and statistical information is reported in Tables 4 to 6. Information on the state of radioactive equilibrium of DL-1a is presented and the values of radium-226 activity determined by two laboratories are reported in Table 7. Analytical results for iron, sulphur and lead are summarized in Table 8 for the convenience of the user of DL-1a.

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 were not used in subsequent computations to avoid possible biasing of the statistics.

HOMOGENEITY TESTS USING INTERLABORATORY RESULTS

Table 5 gives the means and coefficients of variation of each set of results and also the results of the t-tests of differences between the two bottles for each participant at the 5% significance level. Rejection of the null hypothesis of no difference between bottle means is denoted by the code REJECT. For uranium, there was no between-bottle difference detected. For thorium, 2 out of 18 sets of results indicated rejection of the null hypothesis; this 11% rejection rate was typical of previous CCRMP ore certification programs. It must be pointed out however that Lab 12 also reported a second set of results by a different method - X-ray fluorescence - which did not reject the null hypothesis.

The degree of homogeneity of DL-1a is also illustrated in Fig. 1 in which the difference between the means for the two bottles is plotted against the overall mean of the results for both bottles for each set of results. 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)

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

where 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 $\bar{x}..$ by

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

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

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

The value of ω^2 is estimated by

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

where

$$s_2^2 = \sum_i^k n_i \, (\bar{x}_i. - \bar{\bar{x}}..)^2 \Big/ k-1$$

The variance of the overall mean is given by

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

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

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

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.

DISCUSSION OF ANALYTICAL RESULTS

Table 3 is a summary of a methodological classification of accepted analytical results where there is a clear-cut distinction between types of methods in decomposition, separation and determinative steps. As expected, the optical fluorimetric and colorimetric method were the most frequently used methods for uranium and thorium, respectively. Neutron activation analysis was also popular. An attempt to detect a statistial significance between the overall means based on type of method was not performed. Most of the method categories were used by an insufficient number of laboratories, N, to warrant study.

STATE OF SECULAR EQUILIBRIUM OF DL-1a

The state of the secular equilibrium of DL-1a was investigated by Lab 32. The activity ratio of U-234 to U-238, as determined by high resolution alpha spectrometry, was 0.983, indicating DL-1a to be approximately 2% depleted in U-234. The ratio of the activities of Th-228 to Th-232 and of Th-230 to Th-232 were also determined by high resolution alpha spectrometry and found to be 0.99 and 4.85, respectively. The former indicates a high degree of equilibrium in the thorium chain. The latter is somewhat higher than the value of 4.70 which can be calculated using the ratio of the half lives of Th-232 and U-238 and the concentrations of U and Th in DL-1a. Conversion of Ra-226 values to the U equivalent gives a value of 0.0103% which in comparison with the certified value of 0.0116% U suggests that the U chain is in close equilibrium down to Ra-226.

Table 3 - Recommended values and associated statistical parameters (outliers excluded)

Element	No. of laboratories	No. of results	Overall mean	95% CL low	95% CL high	σ_A*
			wt %			
U	28	286	0.0116	0.0113	0.0119	0.0004
Th	18	187	0.0076	0.0072	0.0080	0.0003

*Average within-set standard deviation

Table 4(a) - Summary of analytical methods for uranium (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	$\bar{x}$ (wt %)
Fluorimetric	HNO_3 + one or more of HCl, HF, $HClO_4$; no details on pellet preparation	8	2,5,12, 17,20,24c, 28,29	102	0.0117
	HNO_3 + HCl + $HClO_4$; U extracted with tetrapropylammonium nitrate and methylisobutyl ketone; fused with Na_2CO_2-NaF	1	8b	10	0.0115
	HNO_3 + HF; aliquot fused with Na_2CO_3 + K_2CO_3 + NaF	1	18	10	0.0120
	Acid decomposition, no details; U separated by ion-exchange; fused with NaF	1	23	10	0.0101
Titrimetric	HNO_3 + HCl + HF + $HClO_4$; U (VI) reduced to U (IV) by Fe (II); titrated with $K_2Cr_2O_7$	4	1a,1b,7a, 7b	40	0.0118
Colorimetric	HNO_3 + HF + $HClO_4$ (+ H_2SO_4); U extracted into tri-octylphosphine oxide; U determined as 2-(5-bromo-2-pyridylazo)-5-diethylaminophenolate	2	4,16	20	0.0112
	HNO_3; U extracted with ethylacetate; hydrogen peroxide-sodium hydroxide, differential finish	1	24a	4	0.0122
	HNO_3 + one or both $HClO_4$, HF; U extracted with ethylacetate; hydrogen peroxide-sodium hydroxide, differential finish	2	24b,24d	10	0.0121
X-ray fluorescence	Somar binder for pellet preparation	1	21	10	0.0115
	No details for pellet preparation	1	12a	10	0.0113
Neutron activation	Measurement of delayed neutron emission after irradiation with thermal neutrons	4	8a,15b, 27,30	48	0.0113
	Measurement of ^{239}U 74.66-Kev γ-ray after irradiation with epithermal of neutrons (instrumental technique)	1	15a	10	0.0123
Radiometric	Gamma counting of 1120.3-Kev Bi^{214} after sample sealed in metal can for 21 days	1	32	2	0.0112

Table 4(b) - Summary of analytical methods for thorium (outliers excluded)

Method	Decomposition, separations, etc.	N	Lab No.	n	$\bar{x}$ (wt %)
Colorimetric	Na_2O_2 fusion taken up in dilute HCl; Th separated by ion-exchange on Zerolit 225; thoron	2	3a,3b	20	0.0085
	HNO_3 or HNO_3 + HCl + H_2SO_4; Th extracted with thenoyl-trifluoroacetone-xylene; thoron	2	17,28	25	0.0071
	HCl; final solution is dilute HCl + ascorbic acid; thoron	1	12b	10	0.0070
	HNO_3 + HF + $HClO_4$; Th separated by ion-exchange; arsenazo III	1	8b	10	0.0077
	Acid decomposition, no details; Th separated by ion-exchange; arsenazo III	1	23	10	0.0080
	HNO_3, HF, H_2SO_4; Zr, Fe separated by extraction with 10% alamine-336 in toluene; arsenazo III	1	16	10	0.0058
X-ray fluorescence	Somar or polyvinyl alcohol binder	2	18,21	20	0.0078
	No details given	1	12a	10	0.0061
Radiometric	Measurement of 262 MeV line from Tl^{208} after sample was sealed in metal can for 21 days	1	32	2	0.0079
	Measurement of 2.4-2.82 MeV energy lines	1	24	10	0.0077
Neutron activation	Measurement of gamma emission at 10 days after irradiation with thermal neutrons	1	8a	10	0.0082
	Measurement of ^{233}Th 86.9-KeV line after irradiation with epithermal neutrons	1	15a	10	0.0073
	Measurement of gamma emission after irradiation with thermal neutrons	2	15b,30	30	0.0081
Isotope dilution - mass spectrometric	Sample spiked with ^{230}Th; HNO_3 + HF + $HClO_4$; Th separated by ion-exchange; Th isotope ratios by thermal ionization mass spectrometry	1	19		0.0079

Table 5 - Laboratory means, coefficients of variation and summary of t-test on between bottle results for certified constituents

		U (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	.0113	.0001	5	.0113	.0001	A	10	.0113	.0001	.75
LAB- 1	(TITR)	5	.0114	.0000	5	.0114	.0000	A	10	.0114	.0000	.37
LAB- 2	(FLUOR)	THERE ARE MORE THAN 2 BOTTLES							30	.0119	.0003	2.52
LAB- 4	(COLOR)	5	.0113	.0002	5	.0115	.0002	A	10	.0114	.0002	2.02
LAB- 5	(FLUOR)	10	.0122	.0010	10	.0125	.0007	A	20	.0124	.0009	7.09
LAB- 7	(TITR)	5	.0125	.0005	5	.0125	.0003	A	10	.0125	.0004	2.95
LAB- 7	(TITR)	5	.0121	.0004	5	.0122	.0007	A	10	.0121	.0005	4.46
LAB- 8	(NAA)	5	.0114	.0003	5	.0113	.0003	A	10	.0114	.0002	2.14
LAB- 8	(FLUOR)	5	.0115	.0002	5	.0116	.0002	A	10	.0115	.0002	1.74
LAB-12	(XRF)	5	.0114	.0006	5	.0111	.0004	A	10	.0113	.0005	4.68
LAB-12	(FLUOR)	5	.0107	.0004	5	.0102	.0004	A	10	.0104	.0005	4.74
LAB-15	(NAA)	5	.0124	.0001	5	.0122	.0004	A	10	.0123	.0003	2.42
LAB-15	(NAA)	5	.0118	.0003	5	.0118	.0001	A	10	.0118	.0002	1.85
LAB-16	(COLOR)	5	.0110	.0003	5	.0110	.0002	A	10	.0110	.0002	2.16
LAB-17	(FLUOR)	5	.0121	.0011	5	.0118	.0004	A	10	.0119	.0008	6.75
LAB-18	(FLUOR)	5	.0119	.0009	5	.0121	.0014	A	10	.0120	.0011	9.18
LAB-20	(FLUOR)	4	.0119	.0004	4	.0120	.0001	A	8	.0120	.0003	2.19
LAB-21	(XRF)	5	.0115	.0001	5	.0115	.0001	A	10	.0115	.0001	.80
LAB-23	(FLUOR)	5	.0095	.0012	5	.0107	.0012	A	10	.0101	.0013	12.57
LAB-24	(COLOR)	2	.0122	.0001	2	.0122	.0001	A	4	.0122	.0001	.48
LAB-24	(COLOR)	2	.0121	.0001	2	.0120	0.0000	A	4	.0120	.0000	.42
LAB-24	(FLUOR)	2	.0118	0.0000	2	.0119	.0002	A	4	.0118	.0001	1.06
LAB-24	(COLOR)	3	.0121	.0001	3	.0121	.0000	A	6	.0121	.0001	.50
LAB-27	(NAA)	4	.0106	.0004	5	.0105	.0002	A	9	.0105	.0003	2.56
LAB-28	(FLUOR)	5	.0105	.0014	5	.0094	.0012	A	10	.0100	.0013	13.32
LAB-29	(FLUOR)	5	.0121	.0003	5	.0124	.0003	A	10	.0123	.0003	2.47
LAB-30	(NAA)	9	.0114	.0002	10	.0115	.0002	A	19	.0114	.0002	1.61
LAB-32	(RADIO)	INSUFFICIENT DATA							2	.0112	.0004	3.79

Variance between sets, between bottles and within bottles = 5.17×10^{-7}, 1.04×10^{-8} and 3.43×10^{-7}, respectively.

TABLE 5 (cont'd)

		Th (wt %)									
		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 3	(COLOR)	5	.0093	.0008	5	.0088	.0005	A	10	.0091	.0007	7.65
LAB- 3	(COLOR)	5	.0078	.0004	5	.0080	.0002	A	10	.0079	.0003	3.98
LAB- 8	(NAA)	5	.0083	.0003	5	.0081	.0002	A	10	.0082	.0003	3.33
LAB- 8	(COLOR)	5	.0078	.0001	5	.0076	.0003	A	10	.0077	.0002	2.90
LAB-12	(XRF)	5	.0059	.0007	5	.0062	.0005	A	10	.0061	.0006	9.79
LAB-12	(COLOR)	5	.0065	.0003	5	.0076	.0004	REJECT	10	.0070	.0007	9.43
LAB-15	(NAA)	5	.0072	.0001	5	.0074	.0003	A	10	.0073	.0002	3.35
LAB-15	(NAA)	5	.0076	.0005	5	.0077	.0004	A	10	.0076	.0004	5.70
LAB-16	(COLOR)	5	.0058	.0001	5	.0058	.0001	A	10	.0058	.0001	1.62
LAB-17	(COLOR)	5	.0066	.0004	5	.0067	.0003	A	10	.0067	.0003	4.74
LAB-18	(XRF)	5	.0080	.0004	5	.0079	.0004	A	10	.0080	.0004	4.75
LAB-19	(ID)	5	.0080	.0002	5	.0079	.0002	A	10	.0079	.0002	2.65
LAB-20	(XRF)	2	.0083	.0004	2	.0108	.0004	REJECT	4	.0095	.0015	15.49
LAB-21	(XRF)	5	.0076	.0001	5	.0076	.0002	A	10	.0076	.0001	1.84
LAB-23	(COLOR)	5	.0081	.0006	5	.0078	.0005	A	10	.0080	.0005	6.66
LAB-24	(RADIO)	5	.0077	.0001	5	.0077	.0000	A	10	.0077	.0000	.55
LAB-28	(COLOR)	7	.0072	.0004	8	.0073	.0003	A	15	.0073	.0003	4.45
LAB-30	(NAA)	10	.0084	.0003	10	.0084	.0006	A	20	.0084	.0005	5.36
LAB-32	(RADIO)	INSUFFICIENT DATA							2	.0079	.0003	3.58

Variance between sets, between bottles and within bottles = 6.13×10^{-7}, 3.06×10^{-8} and 1.42×10^{-7}, respectively.

Table 6 - Analytical results for reference concentrate DL-1a

REFERENCE ORE DL-1A

URANIUM (WT %)

LAB- 2 (FLUOR)	.0122	.0117	.0121	.0115	.0124	.0121	.0121	.0123	.0117	.0120
	.0119	.0116	.0124	.0120	.0121	.0122	.0115	.0114	.0120	.0120
LAB- 5 (FLUOR)	.0116	.0123	.0123	.0121	.0117	.0117	.0115	.0115	.0120	.0119
	.013	.013	.011	.014	.013	.011	.012	.012	.012	.011
LAB- 8 (FLUOR)	.013	.013	.013	.013	.013	.012	.011	.012	.013	.012
LAB-12 (FLUOR)	.0111	.0116	.0115	.0117	.0115	.0115	.0113	.0117	.0117	.0117
LAB-17 (FLUOR)	.0113	.0110	.0103	.0105	.0105	.0103	.0104	.0095	.0106	.0100
LAB-18 (FLUOR)	.0116	.0136	.0129	.0109	.0115	.0118	.0117	.0123	.0112	.0118
LAB-20 (FLUOR)	.0112	.0127	.0131	.0111	.0116	.0144	.0116	.0108	.0115	.0121
LAB-23 (FLUOR)	.0119	.0114	.0123	.0120	.0121	.0119	.0121	.0119		
LAB-24 (FLUOR)	.0082	.0083	.0100	.0109	.0100	.0088	.0104	.0113	.0118	.0110
LAB-28 (FLUOR)	.0118	.0118	.0120	.0117						
LAB-29 (FLUOR)	.009897	.009887	.010288	.009487	.0129	.009087	.009127	.008726	.008726	.0115
	.01189	.01192	.01214	.01227	.01250	.01199	.01208	.01250	.01250	.01279
LAB- 1 (TITR)	.0111	.0113	.0113	.0112	.0114	.0113	.0112	.0112	.0113	.0113
LAB- 1 (TITR)	.0113	.0114	.0114	.0114	.0114	.0114	.0113	.0114	.0114	.0114
LAB- 7 (TITR)	.0123	.0122	.0128	.0119	.0131	.0123	.0126	.0127	.0121	.0127
LAB- 7 (TITR)	.0121	.0122	.0114	.0121	.0126	.0119	.0124	.0112	.0125	.0130
LAB- 4 (COLOR)	.01147	.01111	.01118	.01165	.01109	.01168	.01161	.01153	.01120	.01140
LAB-16 (COLOR)	.01079	.01081	.01088	.01115	.01144	.01076	.01083	.01104	.01105	.01133
LAB-24 (COLOR)	.0122	.0121	.0121	.0122						
LAB-24 (COLOR)	.0121	.0120	.0120	.0120						
LAB-24 (COLOR)	.0120	.01215	.01205	.0121	.01215	.01205				
LAB-12 (XRF)	.0106	.0109	.0121	.0117	.0117	.0115	.0114	.0104	.0111	.0112
LAB-21 (XRF)	.0116	.0116	.0114	.0115	.0114	.0115	.0114	.0114	.0116	.0114
LAB- 8 (NAA)	.0111	.0118	.0113	.0114	.0115	.0112	.0117	.0115	.0111	.0112
LAB-15 (NAA)	.0124	.0124	.0124	.0126	.0124	.0128	.0121	.0118	.0120	.0121
LAB-15 (NAA)	.0115	.0115	.0122	.0119	.0118	.0119	.0116	.0119	.0119	.0119
LAB-27 (NAA)	.0110	.0103	.0103	.0109	.0106	.0102	.0105	.0106	.0105	
LAB-30 (NAA)	.01135	.01132	.01176	.01156	.01118	.01146	.01112	.01135	.01132	.01161
	.01152	.01134	.01149	.01118	.01168	.01127	.01168	.01152	.01154	
LAB-32 (RADIO)	.0109	.0115								

TABLE 6 (cont'd)

	THORIUM (WT %)									
LAB- 3 (COLOR)	.00912	.01034	.00880	.01002	.00839	.00905	.00856	.00948	.00831	.00855
LAB- 3 (COLOR)	.00811	.00816	.00721	.00803	.00772	.00787	.00794	.00833	.00785	.00820
LAB- 8 (COLOR)	.0079	.0079	.0077	.0077	.0076	.0076	.0071	.0077	.0077	.0077
LAB-12 (COLOR)	.0069	.0065	.0062	.0064	.0064	.0076	.0074	.0072	.0076	.0082
LAB-16 (COLOR)	.00567	.00575	.00579	.00590	.00591	.00568	.00569	.00579	.00584	.00590
LAB-17 (COLOR)	.0062	.0062	.0069	.0069	.0069	.0066	.0066	.0064	.0070	.0070
LAB-23 (COLOR)	.0077	.0075	.0081	.0079	.0091	.0076	.0079	.0086	.0074	.0077
LAB-28 (COLOR)	.0067	.0067	.0076	.0077	.0075	.0073	.0072	.0074	.0072	.0077
	.0074	.0073	.0072	.0068	.0074					
LAB-12 (XRF)	.0054	.0054	.0070	.0062	.0055	.0060	.0069	.0059	.0058	.0066
LAB-18 (XRF)	.0076	.0078	.0086	.0078	.0082	.0073	.0082	.0081	.0082	.0077
LAB-20 (XRF)	.0085	.0080	.0110	.0105						
LAB-21 (XRF)	.0078	.0075	.0075	.0077	.0077	.0076	.0075	.0074	.0078	.0077
LAB- 8 (NAA)	.0081	.0084	.0079	.0083	.0087	.0082	.0081	.0083	.0077	.0082
LAB-15 (NAA)	.00704	.00742	.00728	.00711	.00721	.00770	.00750	.00686	.00741	.00737
LAB-15 (NAA)	.00819	.00687	.00754	.00769	.00778	.00754	.00796	.00762	.00706	.00822
LAB-30 (NAA)	.00765	.00853	.00873	.00854	.00849	.00842	.00884	.00823	.00839	.00839
	.00774	.00824	.00788	.00891	.00765	.00800	.00903	.00918	.00829	.00883
LAB-24 (RADIO)	.0077	.0078	.0077	.0078	.0077	.0077	.0077	.0077	.0077	.0077
LAB-32 (RADIO)	.0081	.0077								
LAB-19 (ID)	.007981	.008144	.008239	.007676	.007934	.007918	.008090	.007580	.007779	.008068

Table 7 - Values of Ra-226

Lab No.	Method	No. of Results	Ra-226 pCi/g	U equivalent (wt %)
7	Na_2CO_3 fusion; Ra co-precipitated with $PbSO_4$; dissolved in DTPA; radon counted after ingrowth period of 3 days	9	36.7	0.0111
32	Fusion - no details; Ra co-precipitated with $BaSO_4$ and measured with high resolution alpha spectrometry	10	34.0	0.0103

Table 8 - Analytical results for iron, sulphur and lead

Element	Lab No.	Method	No. of Results	Overall mean (wt %)
Fe	1	Atomic absorption	3	0.90
	12	Titrimetry ($K_2Cr_2O_7$)	10	0.96
S	1	Combustion	4	0.42
	1	Wet oxidation	2	0.42
	7	Combustion	6	0.370
	12	Combustion	10	0.43
Pb	21	X-ray fluorescence	12	0.641

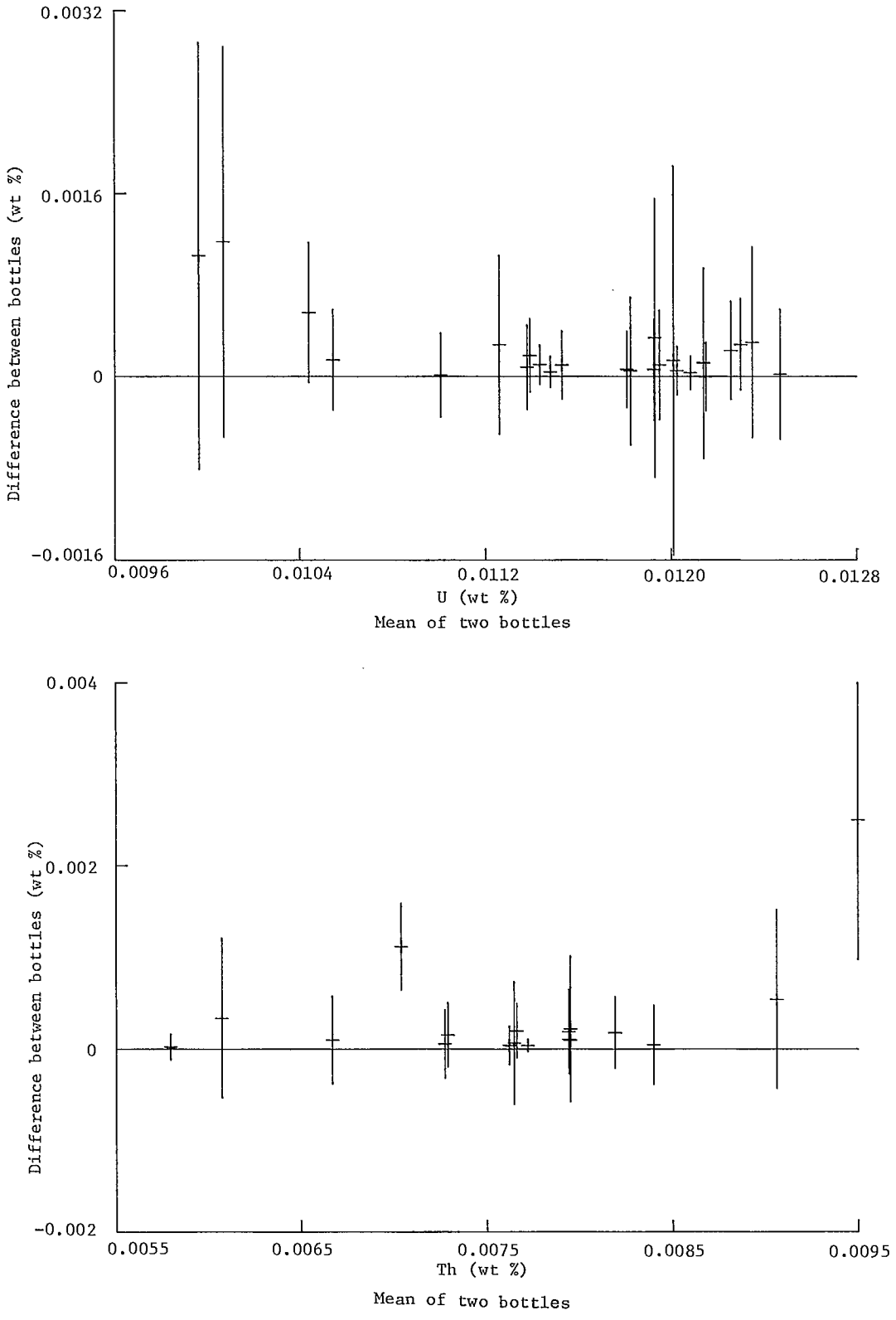

Fig. 1 - Degree of homogeneity of DL-1a. Vertical bars represent 95% confidence intervals for the difference between the means of two bottles for each laboratory

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APPENDIX A

PARTICIPATING LABORATORIES

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Australian Atomic Energy Commission, Nuclear Science and Technology Branch, Lucas Heights, N.S.W., Australia.

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

British Ceramic Research Association, Stoke-on-Trent, Staffordshire, England.

British Columbia Department of Energy, Mines and Petroleum Resources, Victoria, British Columbia.

Canada Centre for Mineral and Energy Technology, Mineral Sciences Laboratories (five independent analysts).

Chemex Labs. Ltd., North Vancouver, British Columbia.

Correlation Laboratories Ltd., Cobden, Ontario.

Denison Mines Ltd., Elliot Lake, Ontario.

Department of Chemistry, Texas A&M University, College Station, Texas.

Department of Physics, University of Calgary, Calgary, Alberta.

Eldorado Nuclear Ltd., Metallurgical Laboratories, Ottawa, Ontario.

Eldorado Nuclear Ltd., Mining Division, Eldorado, Saskatchewan.

Eldorado Nuclear Ltd., Refinery Division, Port Hope, Ontario.

Madawaska Mines Ltd., Bancroft, Ontario.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C.

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

Rio Algom Ltd., Process Development Dept., Elliot Lake, Ontario.

University of California, Los Alamos Scientific Laboratory, Los Alamos, N.M., U.S.A.

University of Vienna, Analytical Institute, Division of Analysis of Nuclear Raw Materials, Vienna, Austria.