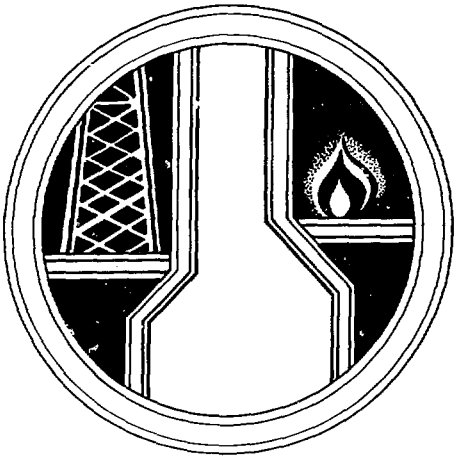
Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

NO. 85-4E

CANMET REPORT



RL-1: A CERTIFIED URANIUM REFERENCE ORE

H.F. STEGER AND W.S. BOWMAN

CANMET
Canada Centre for Mineral and Energy Technology

Centre canadien de la technologie des minéraux et de l'énergie

Canada

CANMET-R--85-4E

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MINERAL RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES

CANMET REPORT 85-4E

JUNE 1985

Available in Canada through

Authorized Bookstore Agents
and other bookstores

or by mail from

Canadian Government Publishing Centre
Supply and Services Canada
Ottawa, Canada K1A 0S9

Catalogue No. M38-13/85-4E Canada: $3.25
ISBN 0-660-11910-2 Other Countries: $3.90

Price subject to change without notice

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by

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

SYNOPSIS

A 145-kg sample of a uranium ore from Rabbit Lake, Saskatchewan, has been prepared as a compositional reference material. RL-1 was ground to *minus* 74 μm and mixed in one lot. Approximately one half of this ore was bottled in 100-g units, the remainder being stored in bulk. The homogeneity of RL-1 with respect to uranium and nickel was confirmed by neutron activation and X-ray fluorescence analytical techniques.

In a "free choice" analytical program, 13 laboratories contributed results for one or more of uranium, nickel and arsenic in one bottle of RL-1. Based on a statistical analysis of the data, the following recommended values were assigned: U, 0.201%; Ni, 185 μg/g; and As, 19.6 μg/g.

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Note: Major contributions were also made by other staff members of the Mineral Sciences Laboratories.

RL-1: MINERAI DE RÉFÉRENCE TYPE D'URANIUM

par

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

SYNOPSIS

Un échantillon de 145 kg de minerai type d'uranium provenant de Rabbit Lake en Saskatchewan a été préparé comme matériau de référence de composition. Le RL-1 a été broyé à une granulométrie de moins 74 µm et mélangé en lot de minerai. Approximativement une moitié de ce minerai a été embouteillée en unités de 100-g; le reste se met en réserve en gros. L'homogénéité du RL-1 quant à l'uranium et au nickel a été confirmée par des méthodes d'activation neutronique et de fluorescence X.

En vertu d'une campagne analytique de "libre choix", 13 laboratoires ont soumis des résultats pour un ou plusieurs des éléments suivants: uranium, nickel et arsenic sur une bouteille du RL-1. Suite à l'analyse statistique des données, les valeurs recommandée suivantes ont été assignées: U, 0,201 %; Ni, 185 µg/g; et As, 19,6 µg/g.

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Nota: D'autres membres du personnel des Laboratoires des sciences minérales ont également apporté une grande contribution à ce projet.

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INTRODUCTION

The preparation, characterization and certification of uranium ore RL-1 is a further contribution of the Canadian Certified Reference Materials Project (CCRMP) in its endeavour 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).

RL-1 was prepared as a higher uranium-bearing complement to uranium tailings sample UTS-4 previously prepared for the same orebody (2); also, the attempt to characterize nickel in UTS-4 was unsuccessful and it was decided to repeat this using an ore sample.

An interlaboratory program was conducted to obtain results for uranium, nickel and arsenic from 13 commercial, industrial and government laboratories using analytical methods of their choice. The results should therefore be indicative of the practical state-of-the-art of the analysis for these elements.

NATURE AND PREPARATION

The raw material for RL-1 was donated in August of 1984 to CCRMP by Eldor Mines Ltd. of Saskatoon, Saskatchewan. It is typical of the uranium deposit at Rabbit Lake, Saskatchewan. The host rock is a siliceous dolomite that has been highly altered and fractured (3). The orebody consists of a high-grade zone of uranium mineralization in the centre of a brecciated zone, grading to low grade in the lesser brecciated perimeter.

The raw material was dry-ground in September 1984 to pass a 74 µm screen. The powdered ore weighing 145 kg was tumbled in a 570 L conical blender for 12 h and bottled in 100-g units. After the selection of bottles for the confirmation of homogeneity, approximately one half of RL-1 was removed from the bottles and is stored in bulk.

The analysis of 15 randomly selected bottles of RL-1 for uranium by neutron activation analysis and for nickel by x-ray fluorescence analysis demonstrated the material to be sufficiently homogeneous for use as a compositional reference material. The results of the evaluation of the homogeneity of RL-1 are reported in Appendix A.

The chemical composition and particle size analysis of RL-1 are reported in Tables 1 and 2.

Table 1 - Approximate chemical composition

Element	Mass %
Si	25.3
Al	6.5
Fe	2.3
Ca	1.8
Mg	9.2
C, total	0.81
Ti	0.25
K	0.22
Na	0.06
U	0.20
S	0.13
Ni	185 µg/g
As	20 µg/g
L.O.I.	10.2
H_2O (105°C)	0.85

*Mean of a minimum of two determinations.

Table 2 - Particle size analysis (wet screen)

Size of fraction (µm)	wt %
-104 + 74	0.0
-74 + 46	11.6
-46 + 37	8.8
-37	79.6

INTERLABORATORY PROGRAM FOR CERTIFICATION

The laboratories that participated in the certification program are listed in Appendix B. Each was assigned a code number which bears no relation to its alphabetical order. The results from CANMET are reported openly.

Each laboratory was requested to contribute five replicate results for uranium, nickel and arsenic for one bottle of RL-1 by methods of its own choice and to report the results on an "as is" basis. Some laboratories however deviated from the request for five results for an element. When a laboratory submitted results by more than one method for an element, each set was considered statistically independent.

The recommended values for RL-1 are presented in Table 3. Methodological and analytical information is presented in Tables 4 and 5.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

DETECTION OF OUTLIERS

Any sets of results whose means differed by more than twice the overall standard deviation from the initially calculated mean value were not used in subsequent computations to avoid biasing of the statistics. Also, sets of results considered to have relatively high variance were rejected. All results that were rejected are identified in Tables 5a through 5c.

ESTIMATION OF CONSENSUS VALUES AND 95% CONFIDENCE LIMITS

A one-way analysis of variance technique was used to estimate the consensus value and 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 (4).

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

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 the 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

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

where

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

The variance of the overall mean is given by

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

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

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

The results of the testing of the homogeneity of RL-1 were included. However, to avoid giving an unduly heavy weighting to the contribution for uranium, only five results were selected at random out of the 45 available.

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.

CRITERION FOR CERTIFICATION

The ratio of the between-laboratory to the within-laboratory standard deviation, σ_B/σ_A, where

$$\sigma_B = \sqrt{\left[\sum_i^k \left(\bar{x}_i. - (\sum_i^k \bar{x}_i.)/k\right)\right]^2 \quad k-1}$$

is a measure of the quality of the certification data for the reference materials of CCRMP (5). The acceptable upper limit for σ_B/σ_A is 3 for all elements except uranium for which an upper limit of 2 is more realistic.

The criterion for the certification of an element in a reference material is RP, the percentage of sets of results that must be rejected to give a value of σ_B/σ_A equal to or less than the acceptable upper limit. RP should not exceed 15%.

The values of σ_B/σ_A and RP for RL-1 are reported in Table 6. RL-1 meets the certification criterion of RP < 15% for $\sigma_B/\sigma_A \leq 2$ as required for uranium.

DISCUSSION

Table 4 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 determination steps. No attempt was made for any element to detect a statistically significant difference between the overall means of the more popular methods because there was generally not a sufficient number to warrant the test.

Figure 1 illustrates the plot of the relative frequency of occurrence against the concentration intervals for uranium, nickel and arsenic. The observed distributions show the consensus attained by the participating laboratories.

A comparison of the nickel value and 95% confidence intervals for RL-1 at 185 ± 5 µg/g with that for UTS-4 (a tailings sample prepared from the ore which RL-1 was prepared) at 151 ± 26 µg/g demonstrates an appreciable increase in the quality of the consensus for this element.

PROCEDURE FOR CHECKING AN ANALYTICAL METHOD USING RL-1 (6)

Perform n replicate determinations (from separate sub-samples) using the analytical method that is being tested. It is suggested that n = 10 for a one-time investigation. For a periodic check of accuracy of an analytical method, n = 2 for each period is sufficient; however, the total number of replicates should be greater than 10.

Compute the following statistics

$$\bar{X} = \sum_{i=1}^{n} X_i/n \text{ - mean}$$

$$S_W = \sqrt{\sum_{i=1}^{n} (X_i - \bar{X})^2 \Big/ n-1}$$ - estimated within-laboratory standard deviation i.e., precision of the method

n is the number of analytical results remaining after rejection of outliers.

a) Verification of precision

Compute

$$F = \frac{(S_W)^2}{(S_{rc})^2}$$ where values of S_{rc} for RL-1, the within-laboratories standard deviation, are given in Table 6.

Compare F against $F_o = F_{0.95,\ n-1,\ DF_c}$ obtainable from any statistics book. If the degree of freedom DF_c is not given in the certificate, use $DF_c = 60$.

$F \leq F_o$: the analytical method is sufficiently precise

$F > F_o$: the analytical method is not as precise as those used for certification of RM

b) Verification of accuracy

$$\text{If } \left| \bar{X} - A_c \right| \leq 2\ S_{Lc}$$

then the analytical method has sufficient accuracy. Otherwise, it is not considered to be as accurate as the laboratories accepted in the certification program.

Values of A_c for RL-1 are presented as the "overall mean" in Table 3 and values of between-laboratories standard deviation, S_{Lc}, are reported in Table 6.

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Table 3 - Recommended value and statistical parameters (outliers excluded)

Element	No. of laboratories	No. of sets of results	No. of results	Overall mean	95% CL Low	95% CL High	s_A
U	10	13	67	0.201%	0.195	0.206	0.004
Ni	11	12	61	185 µg/g	180	190	4
As	11	12	60	19.6 µg/g	18.5	20.7	0.9

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

Method	Decomposition, separation, etc.	Lab No.	n	$\bar{x}$ (mass %)
Fluorimetric	One or more of HCl + HNO_3 + HF + H_2SO_4; uranium extraction with ethyl acetate	7, 8	10	0.199
	One or more of HCl + HNO_3 + HF + H_2SO_4; no details on separation if practised	1, 6, 9, 12	22	0.205
X-ray fluorescence	Sample mixed with binder before pelletization	3b, 4	10	0.208
	$Li_2B_4O_7$ + H_3BO_3 fusion; ground up and pelletized	11a	5	0.198
Neutron activation analysis	Delayed neutron counting	3a, 5a, 5b	15	0.201
Colorimetry	HNO_3 + HF + $HClO_4$; residue fused with $NaBF_4$; uranium extracted with TOPO into cyclohexane; color developed with bromo-padap	11b	5	0.199

Table 4b - Summary of analytical methodology for nickel (outliers excluded)

Method	Decomposition, separation, etc.	Lab No.	n	$\bar{x}$ (µg/g)
Atomic absorption spectrometry	One or more of HCl + HNO_3 + HF + $HClO_4$ + H_2SO_4; taken either to dryness or fumes of $HClO_4$ or H_2SO_4; dissolved in dilute HCl or HNO_3	1, 2, 5, 8, 9, 10, 11a, 12a	41	185.0
	HCl + HNO_3 + H_2SO_4; taken to dryness; Ti, Al complexed with HF; $CaSO_4$ filtered off; nickel extracted with dimethylglyoxime into CCl_4; stripped with 20% HCl	CANMET	5	177.7
	Li_2CO_3-H_3BO_3 fusion; taken up in dilute HNO_3	7	5	182.0
DCP-AE spectrometry	HNO_3 + HF + H_2SO_4; taken to dryness; dissolved in dilute HCl + HNO_3	?	5	191.4
Colorimetry	HNO_3 + HF + $HClO_4$; residue fused with $NaBF_4$; color developed with dimethylglyoxime	11b	5	189.?

Table 4c - Summary of methodology for arsenic (outliers excluded)

Method	Decomposition, separation, etc.	Lab No.	n	Mean (ppm)
Flameless atomic absorption spectrometry	One or more of $HCl + HNO_3 + HClO_4$; arsenic reduced to evolved arsine	2a, 5, 7	15	2.5
	NaOH fusion; taken up in dilute acid; arsine formation	6	1	2.7
Colorimetry	One or more of $Br_2 + HCl + HNO_3 + HBr + HClO_4 + H_2SO_4$; taken to dryness; dissolved in dilute HCl or HNO_3; arsenic reduced and evolved as arsine trapped in silver diethyldithiocarbamate in pyridine or CCl_4	1, 2, 3, 4	20	2.5
	$Br_2 + HNO_3 + HCl + H_2SO_4$; arsenic coprecipitated with $Fe_2O_3 \cdot n H_2O$ twice; arsenic oxidized to As(V) and dehydrated silica filtered off; As(V) reduced to As(III) with $FeSO_4$ and extracted into CCl_4 with xanthate; oxidized to As(V) and stripped with water; determined as molybdenum blue complex	CANMET	6	2.5
	$Br_2 + HNO_3 + HCl + HBr + HClO_4$; arsenic extracted into benzene; stripped with water and oxidized to As(V) with $KBrO_3$; determined as molybdenum blue complex	11a	5	2.7
	No details except "Stain Method"	4	6	1.1
Neutron activation analysis	Instrumental neutron activation	8b	6	2.3

Table 5a - Analytical results, laboratory means and standard deviations for uranium

URANIUM, MASS %

						MEAN	S.D.
LAB- 1	(FLUOR)	0.21	0.22	0.20	0.22	0.19	0.208	0.013
LAB- 3	(NAA)	0.1995	0.1987	0.1991	0.1970	0.1977	0.198	0.001
LAB- 3	(XRF)	0.220	0.220	0.212	0.216	0.216	0.217	0.003
LAB- 4	(XRF)	0.2000	0.2000	0.2003	0.1998	0.1998	0.200	0.000
LAB- 5	(NAA)	0.204	0.199	0.203	0.202	0.208	0.203	0.003
LAB- 5	(NAA)	0.203	0.202	0.201	0.208	0.200	0.203	0.003
LAB- 6	(FLUOR)	0.2180	0.2167	0.2174	0.2173	0.2198	0.218	0.001
LAB- 7	(FLUOR)	0.184	0.166	0.208	0.194	0.189	0.188	0.015
LAB- 8	(FLUOR)	0.184	0.189	0.189	0.189	0.194	0.189	0.004
LAB- 9	(FLUOR)	0.199	0.201	0.204	0.202	0.204	0.203	0.002
		0.204	0.204					
LAB-11	(XRF)	0.1984	0.1976	0.1984	0.1976	0.1976	0.198	0.000
LAB-11	(COLOR)	0.19	0.19	0.19	0.19	0.19	0.190	0.000
LAB-12	(FLUOR)	0.190	0.185	0.188	0.200	0.195	0.192	0.006

Table 5b - Analytical results, laboratory means and standard deviations for nickel

	NICKEL, UG/G					MEAN	S.D.
CANMET (AA)	178.2	178.3	175.7	177.5	178.6	177.7	1.2
LAB- 1 (AA)	200.	200.	196.	200.	188.	196.8	5.2
LAB- 2 (AA)	185.	180.	184.	192.	190.	186.2	4.8
LAB- 3 (DCP-AES)	191.	190.	190.	191.	195.	191.4	2.1
LAB- 5 (AA)	174.	172.	172.	172.	171.	172.2	1.1
LAB- 6 (AA)*	223.	232.	223.	213.	213.	220.8	8.0
LAB- 7 (AA)	177.	183.	185.	182.	183.	182.0	3.0
LAB- 8 (AA)	176.	175.	174.	173.	173.	174.2	1.3
LAB- 9 (AA)	184.	182.	184.	195.	205.	191.7	9.7
	200.						
LAB-10 (AA)	194.	190.	194.	184.	184.	189.2	5.0
LAB-11 (AA)	178.	181.	184.	181.	177.	180.2	2.8
LAB-11 (COLOR)	180.	193.	201.	188.	183.	189.0	8.3
LAB-12 (AA)	190.	190.	180.	190.	190.	188.0	4.5
LAB-12 (AA)*	162.	160.	160.	158.	160.	160.0	1.4

*Outlying set.

Table 5c - Analytical results, laboratory means and standard deviations for arsenic

	ARSENIC, UG/G					MEAN	S.D.
CANMET (COLOR)	20.0	19.7	19.8	19.6	19.8	19.8	0.1
LAB- 1 (COLOR)	20.	21.	19.	19.	17.	19.2	1.5
LAB- 2 (COLOR)	18.8	19.0	19.2	20.3	18.1	19.1	0.8
LAB- 3 (AA)	19.7	20.0	20.0	20.0	19.7	19.9	0.2
LAB- 3 (INAA)	20.4	20.4	19.5	20.5	20.8	20.3	0.5
LAB- 5 (AA)	21.	20.	21.	21.	21.	20.8	0.4
LAB- 7 (AA)	16.0	18.3	16.7	16.2	15.7	16.6	1.0
LAB- 8 (AA)	21.5	22.0	22.0	22.0	22.5	22.0	0.4
LAB- 9 (COLOR)	15.4	18.0	15.0	17.0	20.0	17.1	2.0
LAB-10 (COLOR)	19.	20.	19.	18.	16.	18.4	1.5
LAB-11 (AA)*	24.	29.	28.	19.	25.	25.0	3.9
LAB-11 (AA)*	26.	31.	28.	24.	26.	27.0	2.6
LAB-11 (COLOR)	23.	22.	21.	22.	22.	22.0	0.7
LAB-12 (COLOR)	20.	19.	20.	20.	21.	20.0	0.7

*Outlying set.

Table 6 - Values of σ_B/σ_A and RP for RL-1

Element	σ_B/σ_A	RP (%)	S_{rc}	S_{Lc}
U	1.8	7.7	0.006%	0.0092%
Ni	2.6	7.1	5.0 µg/g	7.3 µg/g
As	2.4	0.0	1.0 µg/g	1.6 µg/g

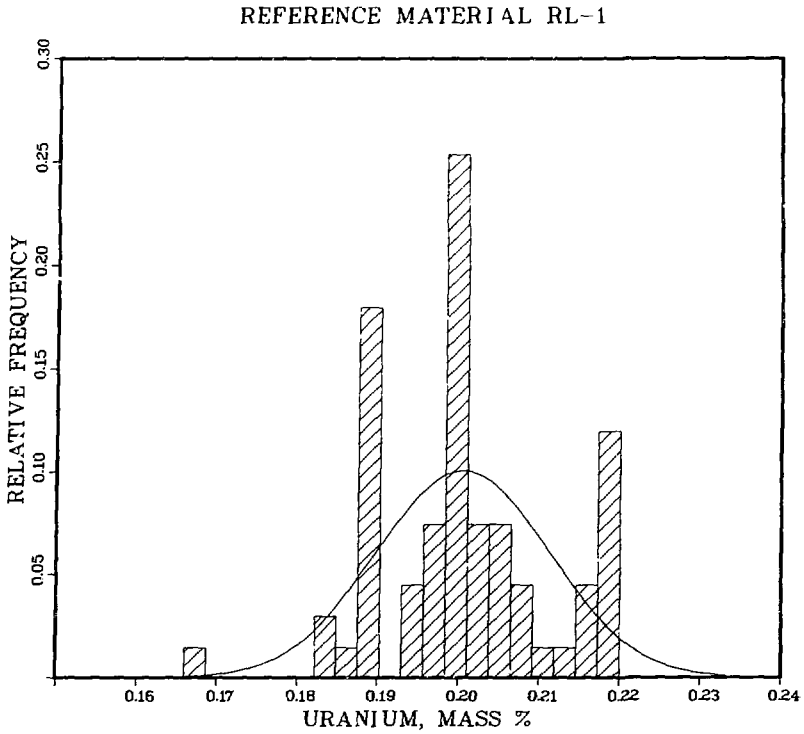

Fig. 1a - Histogram for uranium

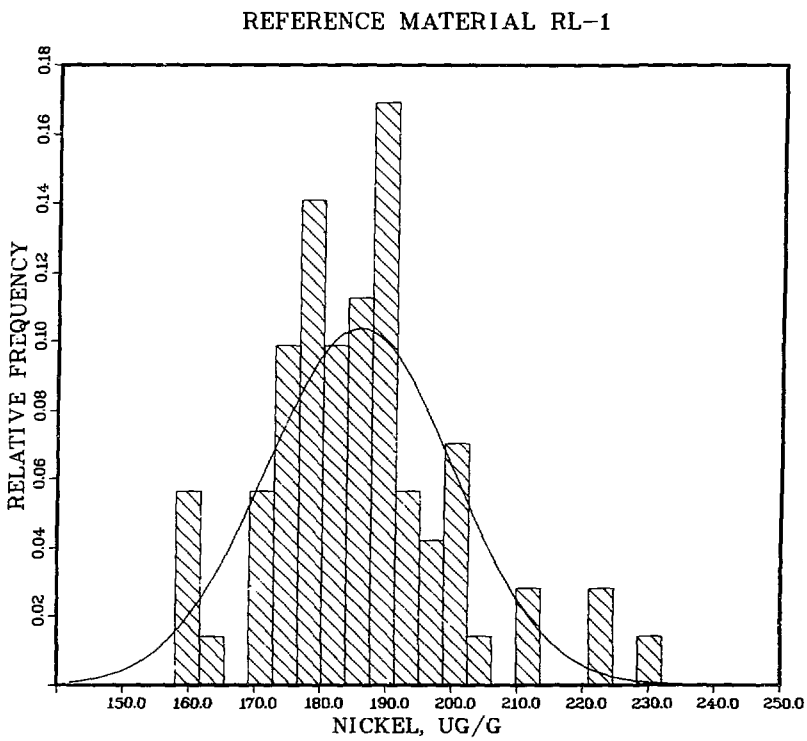

Fig. 1b - Histogram for nickel

REFERENCE MATERIAL RL-1

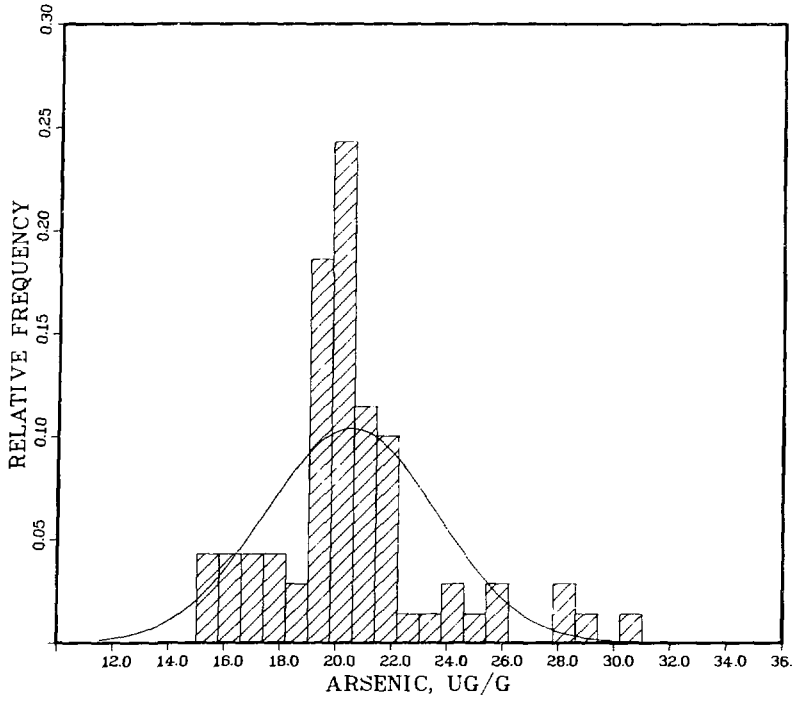

Fig. 1c - Histogram for arsenic

APPENDIX A

CONFIRMATION OF HOMOGENEITY

CONFIRMATION OF HOMOGENEITY

The homogeneity of RL-1 was assessed with respect to uranium by neutron activation analysis by Chemex Laboratories Limited, North Vancouver, British Columbia (Contract #I8510490) and with respect to nickel by X-ray fluorescence analysis at CANMET by analyzing in triplicate 15 bottles selected from a stock of 1366 bottles. The stock was divided into 14 lots of 90 bottles and a 15th lot of 108 bottles. The code number of the first bottle was selected at random out of the first lot. The code number of the remaining bottles selected was given by the code number of the preceding bottle plus 90. The results are shown in Tables A7 to A8. No evidence of any between-bottles inhomogeneity was detected.

Table A7 - Confirmation of homogeneity of RL-1 for uranium (NAA)

	%U			
Bottle No.	Individual			Mean
61	.204	.204	.207	.2052
151	.205	.201	.199	.2018
241	.203	.210	.202	.2047
331	.202	.204	.203	.2027
421	.202	.204	.199	.2015
511	.203	.201	.205	.2030
601	.199	.198	.199	.1990
691	.201	.200	.201	.2007
781	.203	.203	.204	.2030
871	.203	.201	.207	.2035
961	.202	.204	.206	.2041
1051	.206	.203	.201	.2032
1141	.203	.200	.206	.2030
1213	.205	.204	.207	.2058
1277	.199	.204	.201	.2015
			Overall mean is	.2028

Analysis of variance

Source of variation	Degrees of freedom	Sum of squares	Mean squares
Between-sets	14	1.308×10^{-4}	9.342×10^{-6}
Within-sets	30	1.577×10^{-4}	5.257×10^{-6}
Total	44	2.885×10^{-3}	

Calculated F statistic = 1.777

F.95(14,30) = 2.037

Null hypothesis of no difference between bottles is accepted for uranium

Table A8 - Confirmation of homogeneity of RL-1 for nickel (Xrf)

	Counts			
Bottle No.	Individual			Mean
61	315	326	321	320.7
151	320	317	325	320.7
241	322	320	315	319.0
331	320	320	318	319.3
421	313	326	319	319.3
511	315	321	318	318.0
601	325	323	325	324.3
691	326	321	326	324.3
781	316	321	315	317.3
871	324	326	321	323.7
961	318	323	321	320.7
1051	317	326	327	323.3
1141	317	317	319	317.7
1231	327	317	315	319.7
1277	317	323	326	322.0
		Overall mean is		320.7

Analysis of variance

Source of variation	Degrees of freedom	Sum of squares	Mean squares
Between-sets	14	2.380×10^2	17.00
Within-sets	30	4.780×10^2	15.93
Total	44	7.160×10^2	

Calculated F statistic = 1.067

F.95(14,30) = 2.037

Null hypothesis of no difference between bottles is accepted for nickel

APPENDIX B

PARTICIPATING LABORATORIES

Ankara Nuclear Research and Training Centre
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Atlantic Analytical Services Ltd.
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Hudson Bay Mining and Smelting Company Ltd.
Flin Flon, Manitoba
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