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NIOBIUM ORE OKA-1 A CERTIFIED REFERENCE MATERIAL

H.F. STEGER AND W.S. BOWMAN

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES



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OKA-1: A CERTIFIED NIOBIUM REFERENCE ORE

by

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

SYNOPSIS

A 287-kg sample of a niobium ore, OKA-1, from Oka, Quebec, was prepared as a compositional reference material. OKA-1 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" round-robin analytical program, 22 laboratories contributed results for niobium in each of two bottles of OKA-1. A statistical analysis of the data gave a recommended value of $0.37 \pm 0.01\%$ for niobium.

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

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

OKA-1: MINERAL DE RÉFÉRENCE CERTIFIÉ DE NIOBIUM

par

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

SYNOPSIS

Un échantillon de 287 kg de minerai de niobium, OKA-1, provenant d'Oka, Québec, a été préparé comme matériau de référence de composition déterminée. L'OKA-1 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 fluorescence X et par autres méthodes chimiques et embouteillé en unités de 200 g.

En vertu d'un programme analytique de "libre choix", 22 laboratoires ont soumis les résultats sur chacun des deux flacons d'OKA-1 pour le niobium. L'analyse statistique des données a donné une valeur recommandée de $0,37 \pm 0,01\%$ pour le niobium.

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

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

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INTRODUCTION

The preparation, characterization and certification of the niobium ore OKA-1 is another facet of the continuing endeavour of the Canadian Certified Reference Materials Project (CCRMP) to provide compositional references ores, concentrates and related products typical of Canadian deposits and not, in general, available from other sources for use in analytical laboratories associated with mining, metallurgy and the earth sciences. Other reference materials certified by CCRMP are described in a catalogue available from CANMET, Energy, Mines and Resources Canada, Ottawa, (1).

OKA-1 was chosen to serve as a reference material for use in the analytical laboratories associated with the niobium mining industry. An interlaboratory program was conducted to obtain results for niobium from 22 laboratories using analytical methods of their choice. The results should therefore be representative of the current "state-of-the-art" of the analysis of niobium in commercial, industrial and government establishments.

NATURE AND PREPARATION OF OKA-1

The raw material for OKA-1 was donated to CCRMP in October 1974 by St. Lawrence Columbium and Metals Corp. and is typical carbonatite ore from Oka, Quebec. It is essentially a coarse crystalline calcite with minor dolomite, strontianite, siderite, biotite, clay minerals, pyrochlore and sulphide minerals.

OKA-1 was dry-ground in January 1978 to pass a 74- μ m screen. The powdered ore weighing approximately 287 kg was tumbled in a 570-L conical blender for 10 h and then sampled systematically for analysis for niobium by X-ray fluorescence and chemical methods. OKA-1 was found sufficiently homogeneous to qualify as a reference material and was bottled in 200-g units. The approximate mineral and chemical composition and particle size analysis are given in Tables 1 to 3.

Table 1 - Approximate mineralogical composition of OKA-1

Mineral	wt % ^a
Calcite	84
Apatite	5
Biotite	2
Feldspar	2
Magnetite	2
Dolomite	1
Siderite	1
Clays	1
Chlorite	1
Pyrochlore	<1

a - Semi-quantitative estimation by X-ray diffraction

Table 2 - Approximate chemical composition of OKA-1

Element	wt % ^a
Calcium	31.3
Iron	2.8
Silicon	2.4
Magnesium	1.3
Manganese	1.1
Strontium	1.0
Phosphorus	1.1
Aluminum	0.9
Sulphur	0.6
Niobium	0.4
Potassium	0.3
Sodium	0.2
Zinc	0.05
L.O.I.	31.9

a - Mean of a minimum of two determinations

Table 3 - Particle size analysis (wet screen)

Size of fraction (μ m)	wt % ^a
-104 + 74	3
-74 + 55	9
-55 + 46	6
-46 + 37	3
-37	79

a - Mean of duplicate determinations

INTERLABORATORY PROGRAM FOR CERTIFICATION
OF OKA-1

The laboratories that participated in the certification program for OKA-1 are listed alphabetically in Appendix A. Each was assigned a code number which bears no relation to the alphabetical order of the laboratory names.

Each laboratory was requested to contribute five replicate results for niobium on each of two bottles of OKA-1 by a method of their choice and to report the results on an "as is" basis. Some laboratories however deviated from the request for 10 results or contributed results by more than one method. In the latter instance, each set was considered statistically independent. The recommended value for niobium is presented in Table 4. Statistical and other analytical information is presented in Tables 5 and 6. Methodological information is presented in Appendix B.

Table 4 - Recommended value and associated statistical parameters for niobium in OKA-1

(outliers excluded)	
No. of laboratories	20
No. of results	267
Mean, %	0.37
95% confidence limits: low, %	0.36
high, %	0.38
σ_A , average within-set standard deviation	0.006

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. Also, the 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. All results that were rejected are identified in Table 6.

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 bottles at the 5% significance level. Rejection of the null hypothesis of no difference between bottle means is designated by the code REJECT. For niobium, 4 sets out of 26 were rejected. This rejection rate of 15% is typical of previous CCRMP ore certification programs.

The degree of homogeneity of OKA-1 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 value and its 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 (2)

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

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 $\bar{x}..$:

$$\bar{x}.. = \frac{\sum_i \sum_j x_{ij}}{\sum_i n_i} \quad \text{where } \sum_j x_{ij} = k n_i$$

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

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

The value of ω^2 is estimated by

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

where

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

The variance of the overall mean is given by

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

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 .

DISCUSSION OF ANALYTICAL RESULTS

Appendix B 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. As expected, the X-ray fluorescence method predominated. In particular, the fused pellet technique in which niobium is in a matrix consisting essentially of sodium or lithium and calcium borates gave satisfactory results.

An attempt to detect a statistical significance between the overall means based on type of method was not performed because the other method categories were employed by an insufficient number of laboratories to warrant study.

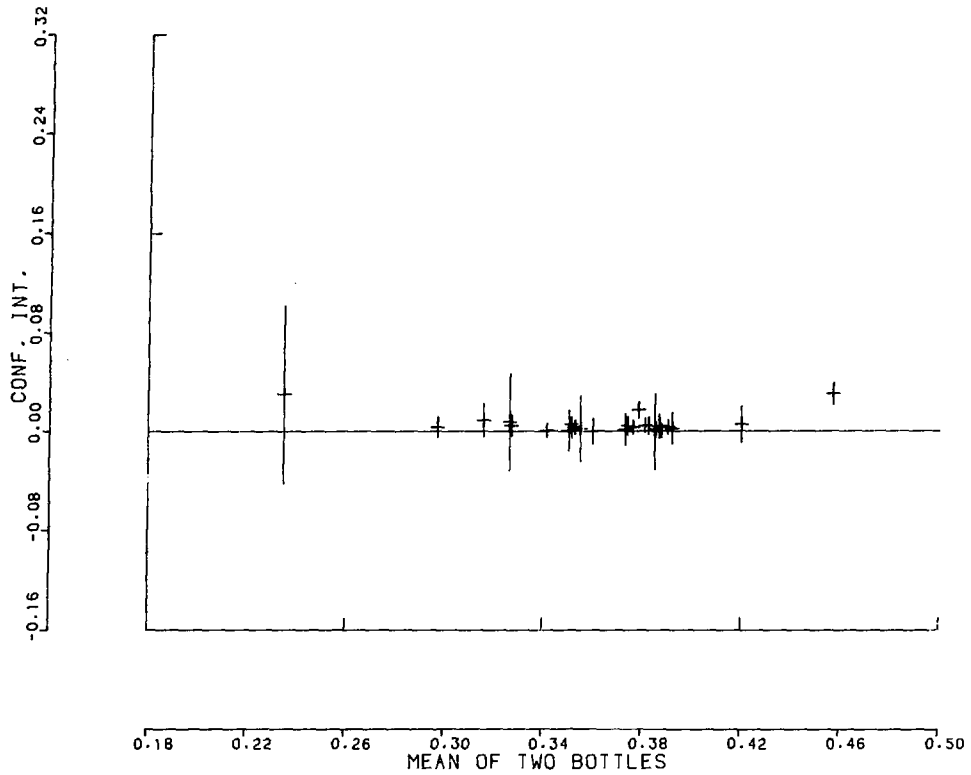
REFERENCE ORE OKA-1
NIOBIUM (WT %)

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

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

	Niobium (wt %)											
	Bottle 1			Bottle 2			Null	Overall				
	N	Mean	St. Dev.	N	Mean	St. Dev.	Hypoth.	N	Mean	St. Dev.	C.V. (%)	
Lab- 1 (Color)	There are more than 2 bottles							30	.3920	.0089	2.26	
Lab- 4 (Color)	16	.3500	.0041	5	.3556	.0063	Reject	21	.3513	.0052	1.47	
Lab- 5 (XRF)	10	.3875	.0025	10	.3877	.0027	A	20	.3876	.0025	.65	
Lab- 5 (XRF)	5	.3888	.0031	5	.3918	.0018	A	10	.3903	.0029	.74	
Lab- 6 (XRF)	6	.3777	.0022	6	.3747	.0018	Reject	12	.3762	.0024	.65	
Lab- 7 (XRF)	5	.3248	.0061	5	.3292	.0058	A	10	.3270	.0061	1.86	
Lab- 8 (XRF)	5	.2954	.0059	5	.2986	.0054	A	10	.2970	.0056	1.88	
Lab- 9 (XRF)	5	.3844	.0049	5	.3804	.0046	A	10	.3824	.0049	1.29	
Lab-10 (Color)	5	.4173	.0064	5	.4227	.0128	A	10	.4200	.0099	2.36	
Lab-11 (Grav)	5	.4722	.0070	5	.4418	.0054	Reject	10	.4570	.0171	3.74	
Lab-12 (AA)	2	.3850	.0071	2	.3850	.0071	A	4	.3850	.0058	1.50	
Lab-12 (AE)	2	.3735	.0035	2	.3725	.0021	A	4	.3730	.0024	.66	
Lab-12 (AE)	3	.3560	.0104	3	.3540	.0125	A	6	.3550	.0104	2.92	
Lab-16 (Grav)	3	.2200	.0100	3	.2500	.0436	A	6	.2350	.0327	13.92	
Lab-16 (AE)	4	.3225	.0310	4	.3300	.0082	A	8	.3263	.0213	6.54	
Lab-18 (Plasma)	5	.3698	.0057	5	.3870	.0032	Reject	10	.3784	.0100	2.66	
Lab-20 (XRF)	5	.3516	.0022	5	.3516	.0027	A	10	.3516	.0023	.66	
Lab-21 (XRF)	5	.3886	.0050	4	.3848	.0024	A	9	.3869	.0043	1.12	
Lab-22 (Plasma)	5	.3788	.0041	5	.3832	.0030	A	10	.3810	.0041	1.09	
Lab-23 (XRF)	7	.3860	.0054	7	.3846	.0049	A	14	.3853	.0050	1.30	
Lab-24 (XRF)	5	.3600	.0071	5	.3600	.0071	A	10	.3600	.0067	1.85	
Lab-25 (Color)	5	.3514	.0047	5	.3542	.0026	A	10	.3528	.0039	1.10	
Lab-25 (Color)	5	.3412	.0040	5	.3418	.0025	A	10	.3415	.0032	.93	
Lab-27 (XRF)	5	.3720	.0045	5	.3760	.0055	A	10	.3740	.0052	1.38	
Lab-28 (XRF)	5	.3500	.0134	5	.3508	.0082	A	10	.3504	.0105	3.00	
Lab-29 (Grav)	5	.3848	.0094	5	.3886	.0020	A	10	.3867	.0067	1.73	
Lab-32 (Color)	5	.3112	.0102	5	.3200	.0079	A	10	.3156	.0098	3.10	

Table 6 - Analytical results for reference ore OKA-1

		Niobium (wt %)									
Lab-12 (AA)		.39	.38	.39	.38						
Lab- 1 (Color)		.38	.39	.38	.39	.40	.37	.39	.39	.39	.39
		.39	.39	.39	.39	.40	.40	.39	.40	.40	.41
		.38	.39	.39	.39	.38	.39	.40	.40	.41	.40
Lab- 4 (Color)		.352	.350	.353	.347	.347	.347	.350	.348	.352	.352
		.352	.358	.354	.340	.350	.345	.356	.363	.346	.354
		.359									
Lab-10 (Color)		.4233	.4200	.4067	.4167	.4200	.4233	.4067	.4367	.4333	.4133
Lab-16 (AE)		.32	.34	*.28	.35	.33	.33	.32	.34		
Lab-25 (Color)		.357	.355	.350	.345	.350	.350	.355	.357	.355	.354
Lab-25 (Color)		.345	.342	.345	.337	.337	.346	.340	.341	.342	.340
Lab-32 (Color)		.326	.301	.304	.317	.308	.315	.320	.325	.330	.310
*Lab-11 (Grav)		.461	.480	.471	.474	.475	.439	.440	.444	.450	.436
*Lab-16 (Grav)		.21	.22	.23	.22	.23	.30				
Lab-29 (Grav)		.3704	.3921	.3802	.3907	.3907	.3858	.3872	.3900	.3893	.3906
Lab- 5 (XRF)		.387	.386	.385	.386	.389	.391	.385	.385	.390	.391
		.387	.386	.390	.389	.392	.384	.385	.385	.390	.389
Lab- 5 (XRF)		.391	.388	.386	.386	.393	.390	.390	.392	.393	.394
Lab- 6 (XRF)		.380	.379	.379	.375	.375	.378	.374	.377	.374	.372
		.376	.375								
Lab- 7 (XRF)		.318	.324	.332	.320	.330	.338	.327	.332	.325	.324
*Lab- 8 (XRF)		.300	.299	.289	.300	.289	.291	.301	.295	.302	.304
Lab- 9 (XRF)		.385	.390	.383	.387	.377	.382	.376	.384	.375	.385
Lab-20 (XRF)		.3502	.3515	.3509	.3554	.3501	.3541	.3495	.3547	.3487	.3510
Lab-21 (XRF)		.393	.394	.389	.384	.383	.383	.385	.383	.388	
Lab-23 (XRF)		.390	.394	.393	.391	.381	.379	.384	.392	.382	.385
		.377	.389	.392	.385						
Lab-24 (XRF)		.35	.37	.36	.36	.36	.36	.36	.37	.35	.36
Lab-27 (XRF)		.38	.37	.37	.37	.37	.37	.38	.38	.38	.37
Lab-28 (XRF)		.3351	.3710	.3512	.3426	.3503	.3393	.3616	.3537	.3521	.3473
Lab-12 (AE)		.376	.371	.374	.371						
Lab-12 (AE)		.344	.361	.363	.364	.340	.358				
Lab-18 (Plasma)		.370	.364	.365	.378	.372	.382	.389	.390	.386	.388
Lab-22 (Plasma)		.382	.384	.376	.378	.374	.380	.386	.384	.386	.380

*Outliers, not used in computations.

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1. Steger, H.F. "Certified reference materials"; CANMET Report 80-6E; CANMET, Energy, Mines and Resources Canada; 1980.
2. Brownlee, K.A. "Statistical theory and methodology in science and engineering"; John Wiley and Sons, Inc., New York; 1960.

APPENDIX A
PARTICIPATING LABORATORIES

APPENDIX A
PARTICIPATING LABORATORIES

ARMCO Inc., Analytical Chemistry, Research and Technology, Middletown, Ohio.

Bonder-Clegg and Co. Ltd., Ottawa, Ontario.

Canada Centre for Mineral and Energy Technology, Chemical Laboratory, Mineral Sciences Division, Ottawa, Ontario.

Ecole Polytechnique, Département de Génie Minéral, Campus de l'Université de Montréal, Montreal, Quebec.

Johannesburg Consolidated Investment Co. Ltd., Mineral Processing Research Laboratory, Knights, South Africa.

London and Scandinavian Metallurgical Co. Ltd., Rotherham, Great Britain.

McLachlan and Lazar (Pty) Ltd., Johannesburg, South Africa.

Metriclab, Ste-Marthe-sur-le-Lac, Quebec.

Ministère de l'Energie et des Ressources, Direction Analyse et Contrôle, Centre de Recherches minérales, Ste-Foy, Quebec.

Newmont Exploration Limited, Metallurgical Department, Danbury, Connecticut.

Niobec, les Services TMG Inc., St.-Honoré, Quebec.

NRC Inc., Analytical and Testing Laboratories, Newton, Massachusetts.

Sandvik Aktiebolag, Coromant Research Centre, Stockholm, Sweden.

Soil Search Lab., Orinda, California.

South Africa Iron and Steel Industrial Corp. Ltd., Research and Process Development, Pretoria, South Africa.

Tantalum Mining Co. of Canada Ltd., Bernic Lake, Manitoba.

Teledyne Wah Chang, Albany, Albany, Oregon.

Texas Instruments Inc., Materials and Process Assurance, Attleboro, Massachusetts.

U.S. Department of the Interior, Bureau of Mines, Reno Metallurgy Research Centre, Reno, Nevada.

U.S. Department of the Interior, Geological Survey, Reston, Virginia.

Union Molycorp, Washington, Pennsylvania.

Universidad de Oviedo, Dr. A. Sanz-Medel, Oviedo, Spain.

APPENDIX B
SUMMARY OF ANALYTICAL PROCEDURES

APPENDIX B

SUMMARY OF ANALYTICAL PROCEDURES

Method	Laboratory No.	Decomposition/Separation	% Nb
X-ray Fluorescence	5	$\text{Na}_2\text{B}_4\text{O}_7$ fusion; Nb_2O_5 standard addition	0.389
	6	$\text{Li}_2\text{B}_4\text{O}_7$ fusion; standard addition	0.376
	7	Pelletized with 2% cellulose acetate suspension; Calibration curve	0.327
	8	Briquetted with sodium alkylaryl-sulphonate as binder; Calibration curve	0.297
	9	1:20 BaO_2 : $\text{Na}_2\text{B}_4\text{O}_7$ fusion; Calibration curve	0.382
	20	15:85 La_2O_3 : $\text{Li}_2\text{B}_4\text{O}_7$ fusion; Calibration curve	0.352
	21	$\text{Li}_2\text{B}_4\text{O}_7$ - ZnO fusion; Calibration curve	0.387
	23	$\text{Na}_2\text{B}_4\text{O}_7$ - SiO_2 - NaBr fusion; Calibration curve	0.385
	24	1:6 BaO_2 - $\text{Li}_2\text{B}_4\text{O}_7$ fusion; Calibration curve	0.360
	27	1:8.5 La_2O_3 : $\text{Li}_2\text{B}_4\text{O}_7$ fusion; Calibration curve	0.374
	28	Pelletized; standard addition	0.350
	Colorimetric	1	$\text{HCl-HF-H}_3\text{PO}_4$ decomp.; HF removed by evaporation; niobium extracted as thiocyanate with ethyl ether from 4M HCl -0.5M tartaric acid medium
4		HCl-HNO_3 decomp.; residue fused with $\text{K}_2\text{S}_2\text{O}_7$ and leached with tartaric acid; Nb determined with PAR as per Belcher et al. Talanta <u>10</u> , 1013 (1963).	0.351
10		Fused with $\text{K}_2\text{S}_2\text{O}_7$; Leached with HCl -tartaric acid; Nb extracted as thiocyanate with amyl alcohol; Nb determined with PAR - See Greenland and Campbell, J. Res. U.S. Geol. Survey <u>2(3)</u> , 353 (1974).	0.420
25		$\text{HCl-HF-H}_3\text{PO}_4$ decomp.; evaporation to paste to remove HF; dissolved in 1M tartaric acid. (a) Nb(V) extracted from tartaric acid- HCl solution in chloroform containing 5,7-dichloroquinolin-8-ol - See A. Sanz-Medel, Rev. Acad. Cienc. (Zaragoza), XXVIII, <u>2(2)</u> , 185 (1973).	0.353
		(b) Aqueous Nb-Sulphochlorophenol S complex - See Cizek and Dolezal, Anal. Chim. Acta. <u>109</u> , 381 (1979).	0.342
32		Fused with KHSO_4 ; leach with HCl -tartaric acid - Nb PAR complex	0.316
DC Plasma	18	$\text{HCl} + \text{HF}$ decomposition; leached with water	0.378

(continued on next page)

Spectrometry	22	Fused with LiBO_2 ; leached with 10% HNO_3 and 3% HF	0.381
Gravimetry	16	Fused with Na_2O_2 -NaOH; leached and acidified with $\text{H}_2\text{SO}_4 + \text{HCl} + \text{HF}$. Passed through column of Dowex AG1-X8, 200-400 mesh; Nb is eluted with 4.5M NH_4Cl -1.5M HF; Nb is precipitated as cupferrate and ignited to Nb_2O_5 .	0.235
	29	HCl decomp.; residue was ignited at 600°C and major part of silica removed with $\text{HNO}_3 + \text{HF}$; the residue was fused with borax - Na_2CO_3 and dissolved in the main filtrate; a cupferron precipitate was made in 10% HCl; redissolved in HNO_3 - HClO_4 ; Nb passed through ion-exchange column and eluted with $\text{HF-NH}_4\text{Cl}$; Nb precipitated as cupferrate and ignited to Nb_2O_5 .	0.316
Atomic Emission	12	(a) $\text{HNO}_3 + \text{HF} + \text{HClO}_4$ decomp.; HClO_4 removed; 1000 ppm NaCl added; 405.9 nm.	0.373
		(b) $\text{HF} + \text{HNO}_3$ decomp.; Same as above	0.355
	16	Fused with 1:1 Na_2O_2 :NaOH; leached with water and neutralized with H_2SO_4 .	0.326
Atomic Absorption	12	$\text{HNO}_3 + \text{HF} + \text{HClO}_4$ decomp.; HClO_4 removed; 1000 ppm NaCl added; 334.4 nm	0.385

