FOREWORD

The International Atomic Energy Agency has, since its beginning, had a continuing interest in making available to its Member States, certified reference materials to enable laboratories engaged in the analysis of nuclear materials, radionuclides and trace elements for which nuclear methods may be used to advantage, so that they can assess and control the quality of their work. Such control is necessary, since the results of these analytical activities are frequently the basis upon which decisions of an economic, administrative, medical or legal nature are made.

Reference materials are provided by the Agency through its Analytical Quality Control Services (AQCS) Programme, which is co-ordinated by the Chemistry Unit at the Seibersdorf Laboratory. The AQCS obtains materials, undertakes their preparation, organizes analytical intercomparisons, certifies reference materials and undertakes the distribution of these materials. Recommendations for new materials are received from other technical divisions within the Agency, from Member State laboratories and through technical meetings. Materials for certification and inclusion in the programme are frequently donated to the Agency by Member State organizations.

The reference materials described herein were prepared in a somewhat different manner. The Division of Nuclear Fuel Cycle noted a requirement for materials specifically designed for use in calibrating laboratory gamma-ray spectrometer instruments used for the analysis of geological and uranium exploration samples. This need was particularly evident in its programme on the intercomparison of calibration facilities for field radiometric equipment and on the construction and use of these facilities. Frequently difficulties were experienced in assigning radioelement grades to calibration facilities due to inadequacies in the analyses carried out on calibration pad materials. These, in turn, were traced to inadequacies of the reference materials used to calibrate the laboratory gamma-ray spectrometer methods.

The Division of Nuclear Fuel Cycle convened a Consultants' Meeting in 1983 to prepare specifications for an appropriate set of reference materials. Following the recommendations of the Consultants' Group, arrangements were made with the Canada Center for Minerals and Energy Technology (CANMET) through its Canadian Certified Reference Materials Programme, to undertake the preparation of a thorium and a uranium reference material of suitable concentration and quality to serve as IAEA AQCS certified reference materials. The result was the completion of RGU-1 and RGTh-1, which together with RGK-1, prepared by the AQCS, form a set of three materials for the calibration of laboratory gamma-ray spectrometers for potassium, uranium and thorium. The preparation of RGU-1, RGTh-1 and RGK-1 is described in this report.

The Agency wishes to thank its Consultants on this project, Dr. R.L. Grasty of the Geological Survey of Canada; Dr. Leif Lövborg of Risø National Laboratory, Denmark, and; Dr. Milan Matolin of Charles University, Czechoslovakia. Thanks are also due to the officers of CANMET, particularly Drs. H.F. Steger and C.W. Smith, for their excellent efforts in the preparation of these reference materials. The Agency officer responsible for the project was A.Y. Smith of the Division of Nuclear Fuel Cycle.

CONTENTS

INTRODUCTION

At a number of meetings and conference over the last ten or fifteen years the need has been expressed for a new set of reference materials specifically designed for the calibration of laboratory gamma-ray spectrometers used for the analysis of geological and other natural materials. In 1983 the IAEA convened a Consultants' Meeting in Vienna to discuss the matter and to provide specifications for such reference materials.

The present Technical Document provides users of these reference materials with information on the recommendations of the consultants' group and on the preparation of RGU-1, RGTh-1 and RGK-1.

CONSULTANTS' MEETING

A consultants' meeting was held in Vienna, December 12 - 14, 1983, to discuss and recommend methods and procedures for the preparation of reference materials for use in gamma-ray assaying of geological materials. The group reported as follows.

The need for carefully prepared, certified reference materials for use in gamma-ray spectrometric analysis of geological materials has been recognized for some years. Presently available materials have a number of defects that limit their usefulness. In the case of uranium bearing materials U and Ra are not in equilibrium and are frequently high in Th. In some cases Ra content is not stated. Thorium reference materials are frequently too high in U and, again, the state of equilibrium is not known. In general, the content of U and Th in these materials is very high, requiring the user to prepare diluted materials, a task difficult for many users to carry out satisfactorily. A further deficiency is that the available materials have generally been calibrated against other reference materials rather than primary standards so that errors in earlier references are carried through to the new ones [1,2,3].

Reference materials for U, Th and K are used for the precise calibration of laboratory gamma-ray spectrometers for the measurement of these radioelements in geological materials. These instruments are, in turn, used to measure the radioelement contents of field instrument calibration facilities. These facilities include large concrete pads for the calibration of airborne gamma-ray spectrometer equipment; small concrete pads for calibration of field gamma-ray spectrometers, and concrete bore-hole models for the calibration of bore-hole logging equipment. Once such calibration facilities are available they are made use of by environmental workers and waste management specialists, as well as uranium explorationists.

A further use of such certified reference materials, of importance in the Agency's Technical Co-operation projects, is in the establishment and calibration of laboratory gamma-ray spectrometer facilities for the analysis of geological materials in developing countries. The lack of satisfactory reference materials has been a handicap in such projects in several countries.

The consultants recommend:

1. That a reference material be prepared for U with a concentration of approximately 400 µg/g ± 8 µg/g U. This should be in secular equilibrium between U and Ra, and be very low in Th and K. This material should be called RGU-1.

2. That a reference material be prepared for Th with a concentration of approximately 800 µg/g ±24 µg/g Th, and very low in U and K. This material should be called RGTh-1.

3. That a reference material be prepared for K with a concentration of approximately 40 % K (by weight). The material should be made from chemical reagent and should be checked for very low content of U and Th. This material should be called RGK-1.

4. Each of the materials should be available in 500 kg quantities, to be distributed in bottles of 500 g. The materials should be distributed as a set of three.

5. Uranium ore (BL-5) available from the Canada Center for Mineral and Energy Technology (CANMET) containing 7.09 % U and in radioactive equilibrium should be used to prepare the U reference material.

6. Thorium material being investigated by the Geological Survey of Canada, if demonstrated to be of suitable quality, should be used to prepare the Th reference material.

7. The IAEA should enter into an agreement with CANMET for the preparation of these two materials, perhaps through a Technical Contract.

8. The K reference material should be prepared by the Agency's Seibersdorf Laboratory from commercially available reagent chemicals.

9. The certification of the U ore already carried out by CANMET should be accepted, the diluted material being checked for final concentration and homogeneity.

10. The certification of the Th ore to be used to prepare the Th reference material should be carried out jointly by CANMET and the Agency through its AQCS Programme.

Consultants: Dr. R. Grasty (Canada)
Dr. L. Lovborg (Denmark)
Dr. M. Matolin (Czechoslovakia)
Dr. O. Suschny (IAEA Laboratory)
Mr. L. Pszonicki (IAEA Laboratory)
Mr. A.Y. Smith (IAEA Scientific Secretary)

Arrangements to begin preparation of these reference materials were made early in 1984 with the Canada Center for Mineral and Energy Technology through an Agency Technical Contract. RGU-1 was the first to be prepared, by dilution with silica sand of CCRMP Uranium Ore BL-5. The certification of BL-5 is described in by Faye, Bowman and Sutarno [4]. The preparation of RGU-1 is described below, reproduced from the report by Steger [5] supplied to the Agency.

The Geological Survey of Canada began the search for a suitable thorium ore material with which to prepare RGTh-1. Suitable material was found at a britholite deposit near Oka, Quebec, and prepared by CANMET under the title OKA-2. Samples of this material were distributed to 29 laboratories by the Agency's AQCS Programme in the spring of 1985. The IAEA programme requested analyses of thorium, uranium, uranium/radium ratio and potassium. At the same time CANMET distributed samples to a selection of laboratories under its Canadian Certified Reference Material Project, requesting analyses for thorium, uranium and lanthanide elements, in keeping with their interest in OKA-2 as a potentially useful compositional reference material particularly for the lanthanide series elements. The results of the two intercomparisons were used by CANMET to determine the recommended values for thorium, uranium, equivalent uranium and potassium. The certification of OKA-2 is described by Smith, Steger and Bowman [6].

RGTh-1 was prepared by dilution with silica sand of OKA-2. The preparation of RGTh-1 is described below, reproduced from a report by Smith, Grasty, Bowman and Steger [7] supplied to the Agency on completion of the work.

RGK-1 was prepared from a batch of potassium sulphate supplied by the Merck Company as Potassium sulphate powder Extra Pure DAC. The content of uranium and thorium was checked by the Agency's Seibersdorf Laboratories.

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THE PREPARATION OF RGU-1, A URANIUM RADIOMETRIC REFERENCE MATERIAL

by

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INTRODUCTION

The Canadian Certified Reference Materials Project (CCRMP) agreed in 1984 to prepare a uranium reference material, RGU-1, on behalf of the International Atomic Energy Agency, IAEA. The work was accepted by the Canada Centre of Mineral and Energy Technology as Cost Recovery Job No. 025202.

RGU-1 is intended for use in calibrating laboratory gamma-ray spectrometers to determine their sensitivities and stripping ratios for determination of U, Th and K ratios. This reference material was to be prepared by dilution of uranium reference ore BL-5 (1) of CCRMP (7.09% U) with a silica matrix of negligible U and Th content to give a final U concentration of 400 ± 8 µg/g. Since the advent of the preparation of RGU-1, BL-5 has been certified for radium-226 (2) and lead-210 (3), thereby confirming BL-5 to be in secular equilibrium. This report describes the physical and chemical procedures by which RGU-1 was prepared and characterized.

PREPARATION AND NATURE

RAW MATERIALS

The characterization and certification of BL-5 are described elsewhere (1-3) - Certificate of Analysis attached. The most important constituent parameters are reviewed in Table 1.

Table 1 - Values of constituents of BL-5

Constituent	Concentration
U	7.09 ± 0.03%*
Ra-226	857 ± 38 Bq/g*
Pb-210	866 ± 21 Bq/g*
Th	40 µg/g
K	0.4%

*Certified value

The silica diluent (500 kg) was purchased from Fisher Scientific, catalogue #S-153. It is a floated powder of approximately 240 mesh size. A particle size analysis is reported in Table 2.

Table 2 - Particle size analysis (wet screen)

Size of fraction (μm)		Silica	RGU-1*
		mass %	
	+104	0.0	0.0
-104	+ 74	8.3	8.3
- 74	+ 46	19.9	19.8
- 46	+ 37	14.0	13.9
- 37		57.8	58.0

*Calculated from corresponding values of silica and BL-5 (1).

The silica was investigated at CANMET by gamma-ray spectrometry and the equivalent uranium and equivalent thorium values were estimated to be 0.2 ± 0.1 and 0.4 ± 0.3 μg/g, respectively. The silica contribution to the overall uranium content of RGU-1 (~400 μg/g) is therefore negligible. On the other hand, the equivalent thorium value of the silica is approximately the same as the 0.2 μg/g Th value expected for RGU-1 from BL-5 (0.004% Th) but does not affect significantly the favorable U:Th ratio required in RGU-1.

The silica was also analyzed by a semi-quantitative optical emission technique.

PHYSICAL PREPARATION

RGU-1 was prepared in two batches because of equipment limitations. For one batch, 245.8 kg of silica were blended with 1.395 kg of BL-5 for 8 h in a 570-L blender. The other batch consisted of 253.8 kg of silica and 1.440 kg BL-5 and was treated similarly. These batches were divided approximately in-half and each portion of one batch was blended with a portion of the other batch for 3 h, thereby giving two new batches. This procedure was repeated twice more to ensure that the two batches obtained at the end were homogeneous. RGU-1 was bottled in 500-g lots in plastic bottles supplied by IAEA. These bottles had narrow necks intended for liquid storage and as a

consequence gave considerable difficulty in the filling process. Material losses were much greater than expected and only 972 instead of 1000 bottles could be provided.

HOMOGENEITY CONFIRMATION

The homogeneity of RGU-1 was assessed by CANMET by an X-ray fluorescence technique and by Chemex Laboratories Ltd., North Vancouver, British Columbia (P.O. No. 4.5277) by a neutron activation technique by analyzing in triplicate for uranium 16 bottles selected from the stock of 972. The stock was divided into 15 lots of 62 bottles and a 16th lot of 42 bottles. The code number of the bottle from the first lot was selected at random out of 1 to 62. The code number of the remaining 15 bottles was given by the code number of the preceding bottle plus 62. Chemex Laboratories Ltd., were sent three 2-g samples from each bottle; the samples were coded accordingly to a random selection scheme.

A one-way analysis of variance technique was used to assess the homogeneity (4). Herein the ratio of the between-bottles to within-bottle mean square is compared to the F statistic at the 95% level of probability. The results for the evaluation by CANMET and by Chemex Laboratories are shown in Tables 3 and 4. No evidence for between-bottles inhomogeneity was detected by either CANMET or Chemex Laboratories Ltd. Another manner of evaluating the homogeneity results is reported in Table 5. Herein the mean of bottles 19 to and including 453 from one batch is compared with that for bottles 515 to and including 949 of the other batch of fully blended material. The observed difference between the mean values is not statistically significant in view of the values of σ, the standard deviation of the means.

NATURE OF RGU-1

The approximate chemical composition of RGU-1 shown in Table 6 is based on the composition of BL-5 and the semi-quantitative optical emission analyses of the silica diluent and RGU-1. The calculated particle size distribution is reported in Table 2.

The uranium concentration of RGU-1 calculated solely on the ratio of silica to BL-5 is 400 ug/g with 95% confidence intervals of $\pm$ 1.7 ug/g. The latter reflects only the estimated uncertainty in the measurement of uranium in BL-5. The potential error in the weighing of the silica was estimated to

Table 3 - Confirmation of homogeneity of RGU-1 by CANMET

Bottle No.	Counts			
	Individual			Mean
19	1097	1078	1064	1079.7
81	1084	1087	1093	1088.0
143	1065	1086	1098	1083.0
203	1052	1078	1086	1072.0
268	1099	1097	1082	1092.7
329	1090	1071	1074	1078.3
391	1075	1078	1070	1074.3
453	1081	1076	1058	1071.7
515	1081	1075	1071	1075.7
577	1079	1082	1069	1076.7
639	1072	1062	1088	1074.0
701	1090	1076	1077	1081.0
763	1054	1066	1053	1057.7
825	1065	1070	1089	1074.7
887	1095	1077	1085	1085.7
949	1067	1064	1074	1068.3
		Overall mean is		1077.1

Analysis of Variance Table

Source of Variation	D.F.	Sum of squares	Mean squares
Between-sets	15	3.084×10^3	2.056×10^2
Within-sets	32	3.737×10^3	1.168×10^2
Total	47	6.822×10^3	

Calculated F statistic = 1.761

F.95(15,32) = 1.992

Null hypothesis of no difference between bottles is accepted for uranium

Table 4 - Confirmation of homogeneity of RGU-1 by Chemex

Bottle No.	Mass % U			
	Individual			Mean
19	.0390	.0390	.0382	.0387
81	.0390	.0390	.0390	.0390
143	.0390	.0382	.0382	.0384
205	.0390	.0382	.0390	.0387
267	.0407	.0382	.0399	.0396
329	.0407	.0382	.0382	.0390
391	.0390	.0399	.0399	.0396
453	.0382	.0399	.0373	.0384
515	.0390	.0390	.0399	.0393
577	.0390	.0390	.0399	.0393
639	.0399	.0390	.0390	.0393
701	.0399	.0390	.0390	.0393
763	.0390	.0390	.0416	.0399
825	.0382	.0382	.0390	.0384
887	.0399	.0390	.0382	.0390
949	.0390	.0399	.0390	.0393
		Overall mean is		.0391

Analysis of Variance Table

Source of Variation	D.F.	Sum of squares	Mean squares
Between-sets	15	8.390×10^{-6}	5.593×10^{-7}
Within-sets	32	2.157×10^{-5}	6.742×10^{-7}
Total	47	2.996×10^{-5}	

Calculated F statistic = 0.830

F.95(15,32) = 1.992

Null hypothesis of no difference between bottles is accepted for uranium

Table 5 - Comparison of means of the two batches of RGU-1

Laboratory	Batch	Bottles	Mean ± σ
CANMET	1	19 - 453	1080.0 ± 7.6 counts
	2	515 - 989	1074.2 ± 8.4 counts
Chemex	1	19 - 453	389 ± 4.7 ug/g U
	2	515 - 989	392 ± 4.2 ug/g U

Table 6

Approximate Chemical Composition of RGU-1

ELEMENT	MASS %
Si	46.4
U	0.04
Al	0.1
Fe	0.03
Ca	0.03
Na	0.02
C, total	0.01
Mg	0.01
Pb	0.008
K	0.002
Ti	0.008
S	0.002
Th	< 1 µg/g

be 1.45 kg for the total mass of 500 kg leading to an uncertainty of ± 1.2 ug/g U. Errors in the weighing of BL-5 were assumed to be negligible. The total uncertainty of the concentration of uranium is composed of the uncertainty of the measurement of uranium in BL-5 and the uncertainty in the dilution by weighing and was estimated to be ± 2.1 ug/g. Therefore:

RGU-1 400 ± 2.1 ug/g U

Analysis of RGU-1 at CANMET by an X-ray fluorescence technique found the concentration of uranium to be 0.041%. The mean from Chemex Laboratories Ltd. was 0.0391% and the overall mean from both laboratories is 0.040%.

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THE PREPARATION FOR IAEA OF RGTh-1, A THORIUM RADIOMETRIC REFERENCE MATERIAL

by

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THE PREPARATION FOR IAEA OF RGTh-1, A THORIUM RADIOMETRIC REFERENCE MATERIAL

by

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ABSTRACT

RGTh-1 was prepared for the International Atomic Energy Agency (IAEA), Vienna, Austria at CANMET in November 1986. It is one of a set of three IAEA reference materials intended for method calibration for radiometric measurements of, respectively, potassium, uranium and thorium in geological samples, particularly by gamma-ray spectrometric methods.

RGTh-1 is a blend of sub-74 µm particles of Canadian Certified Reference Materials Project (CCRMP) reference britholite ore OKA-2 with essentially non-radioactive silica of similar size distribution. The homogeneity of the material was tested by gamma-ray measurement of a sampling representing 5% of the stock of 1012 500-g bottles. Analysis of variance of the data detected no significant evidence of difference between bottles.

Recommended values and uncertainty estimates for thorium, uranium and potassium in RGTh-1 were established using the certified values for these elements in OKA-2, the measured values for the minor contributions from the silica, and the dilution ratio of the preparation. The recommended values are 800.2 ± 15.8 µg/g thorium, 6.26 ± 0.42 µg/g uranium and 0.02 ± 0.01 % potassium, where the uncertainties are 95% confidence interval estimates. The thorium value is certifiable by CCRMP criteria. Since the thorium-232 decay series is demonstrably in secular equilibrium and because of the high thorium/uranium ratio, RGTh-1 is regarded as an acceptable reference material for radiometric measurements of thorium which will complement the IAEA materials RGK-1 and RGU-1 for potassium and uranium respectively.

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TABLES

FIGURES

INTRODUCTION

RGTh-1 is a reference thorium ore material intended for use in the gamma-ray spectrometric analysis of geological materials, primarily for instrument calibration purposes. It is the third of a suite of three to be provided by the International Atomic Energy Agency (IAEA) for the natural radioactive analytes, uranium, potassium and thorium. RGTh-1 was prepared and characterized in accord with IAEA specifications at CANMET in November 1986. This preparation follows, and was similar to the preparation of IAEA reference uranium ore RGU-1 completed at CANMET in 1985(1).

The requirements for RGTh-1 were delineated by an IAEA Consultants' Committee on Gamma-ray Analysis comprising Dr. L. Lovborg of RISO National Laboratory, Denmark, Dr. M. Matolin of Charles University, Czechoslovakia, Dr. O. Suschny and L. Pszonicki of the IAEA Laboratory, Austria, And Dr. R. Grasty of the Geological Survey of Canada, reporting to Dr. A.Y. Smith of IAEA(2). In brief, the requirements were:

- Radiometric equilibrium in the thorium decay series;
- A low potassium content;
- A thorium/uranium ratio in excess of 100;
- A thorium concentration of 800 ± 24 µg/g;
- A 500 kg quantity bottled in 500 g units.

In summer 1984 the Radiation Geophysics Section, GSC undertook the location of suitable mineralogical material for the preparation of RGTh-1. Material from a britholite mineral deposit from the Oka carbonatite near Oka, Quebec was found to have a suitable thorium/uranium ratio in preliminary measurements(3). This material was accepted by the IAEA consultants to be suitable source material for the preparation of RGTh-1. However, since the thorium content was of the order of three percent, homogeneous dilution in a non-radiogenic matrix would be required to obtain a standard suitable for application to geological materials.

At the same time, the Canadian Certified Reference Materials Project (CCRMP) foresaw that the Oka britholite would be a potentially useful high grade reference ore for both thorium and lanthanide elements, which comprise about one third of the matrix as oxides.

The britholite material was subsequently prepared as a compositional reference material with the CCRMP identifier OKA-2 in February 1985 and then distributed to participating laboratories in measurement programs coordinated by IAEA and CCRMP. Results were assessed at CANMET in autumn of 1986 and recommended values for uranium, potassium and thorium were established. RGTh-1 was prepared as a reference material at CANMET in November 1986, and its homogeneity and recommended values for the radiometric elements were established. RGTh-1 was shipped to IAEA in Vienna in December 1986.

This report documents the preparation of RGTh-1 and the establishment of the recommended values for IAEA, as a basis for their certification of this material.

CERTIFICATION OF RADIOMETRIC ELEMENT CONCENTRATIONS IN OKA-2

Thirty laboratories contributed replicated measurements of thorium, uranium, and/or potassium by one or more methods to either the IAEA or the CCRMP measurement projects. The results from the IAEA project were submitted to CCRMP for statistical assessments to compare the results of the two projects and to obtain concentration values certifiable in accord with the consensus.

The preparation, measurement project results and their assessments are fully described in a separate report which may be regarded as an Appendix to this report for purposes of the certification of RGTh-1(4). The overall results are summarized herein.

Results of assessments for thorium, uranium and potassium are presented in Tables 1-3. The recommended values are the mean of all accepted results. Outlier sets were declared either by inspection (sets with means so grossly deviant from the overall means to suspect physical measurement or calculation errors), or on statistical grounds (set means lying beyond two standard deviations of all individuals from the grand mean). The 95% confidence intervals quoted in Tables 1-3 were computed from the analytical data assuming that they follow a random model of one way classification (5).

The measurement results are summarized in Table 5. The CANMET values were from 60 ks measurements of 800 g samples in Marinelli beakers with a 56 cm^3 coaxial Ge spectrometer. Equilibrium thorium and uranium standards of 1 ppm (aqueous solution) and 2 µg/g (BL-4a dispersed in starch), respectively, were used for calibration. The latter material was also used for potassium calibration (5.8 µg/g), assuming zero potassium content of the starch. The sample results are the means obtained from measurements of four photopeaks for thorium (583,911,968 and 2614 KeV), five for uranium (186,352,609,1120 and 1764 KeV) and the single ^{40}K photopeak at 1460 KeV. The GSC measurements were performed on 250 g samples in thin steel cans with spectral acquisition for 6 ks. Backgrounds were measured before and after each sample. Results are based on net intensities in the ^{40}K, ^{214}Bi and ^{208}Tl windows from 1.36-1.55, 1.66-1.85 and 2.43-3.47 MeV respectively. Calibration was performed with thorium and radium reference materials dispersed in a silica matrix as described in reference(7).

Examination of Table 5 shows good agreement between the results obtained by the two laboratories and the values 0.37 ± 0.05 µg/g thorium, 0.22 ± 0.05 µg/g uranium and 0.010 ± 0.005 % potassium were accepted for the silica component contributions to RGTh-1.

PREPARATION OF RGTh-1

RGTh-1 was prepared by mass dilution of OKA-2 with the minus 240 mesh silica to produce a blend having 800 µg/g thorium. Quantities were calculated to yield 525 kg of RGTh-1, sufficient to produce in excess of one thousand 500 gram units.

The blender capacity of about 340 kg required that blending be performed initially in two lots, to be followed by a series of splits and reblendings to ensure homogeneity. Two equal sized lots were initially prepared. For each, 7.2565 ± 0.0019 kg of OKA-2 and 255.2 ± 0.3 kg of silica were weighed and quantitively transferred to the 540L conical blender. The OKA-2 was weighed on a Toledo Model 3710 balance in two lots of 2.5 kg and one lot of 2.0 kg, each to an accuracy of ± 0.6 g and one lot of 256.5 ± 0.1 g on a Mettler top loading balance. The silica was measured in drum lots on a Berkel beam balance to a totalled accuracy of 0.3 kg. The accuracy of each balance was confirmed with standard weights and the

The results from the IAEA and CCRMP measurement sets were initially assessed independently to ascertain the degree of consensus between the programs. Results were then combined to obtain the final recommended values. In the combination, duplicate sets resulting from submissions to both programs were removed.

The results for thorium show excellent agreement between IAEA and CCRMP, and the value from the combined assessment, 2.893 ± 0.057 percent thorium, meets the criteria of CCRMP for certification(5). Treatment of the five gamma-ray radiometric sets (29 results) independently yields 2.885 ± 0.110 % Th, demonstrating that the thorium-232 decay series is in secular equilibrium in OKA-2.

Similarly IAEA and CCRMP agree within their uncertainties for the concentration of uranium in OKA-2. Measurements of equivalent uranium (eU), which are based on measurements of radium-226, or its daughter isotopes, were assessed independently and indicate that the uranium-238 decay series is in secular equilibrium, since eU/U = 0.97 ± 0.17 or 0.98 ± 0.12 by comparison with the IAEA and CCRMP uranium values, respectively. The combined assessment yields 218.6 ± 8.1 µg/g uranium, which is certifiable by CCRMP criteria. The thorium/uranium concentration ratio in OKA-2 is calculated as 132.6 ± 5.6, exceeding the minimum value of 100 specified by the IAEA for RGTh-1.

The approximate composition of OKA-2, compiled from data submitted to the CCRMP measurement program, is given in Table 4.

CHARACTERIZATION OF THE SILICA COMPONENT OF RGTh-1

The silica employed in the synthesis of RGTh-1 was Fisher Scientific Ltd. floated silica powder, about 240 mesh (Fisher nos. 9431560 and 9431561). The supply was purchased in five 100 kg and one 50 kg units each from lot no. 854599.

Gamma-ray measurements were performed at the Chemical Laboratory, CANMET by high-resolution germanium spectrometry and the Airborne Geophysics Section, GSC by dual 5 x 5 inch NaI(Tl) spectrometry to obtain the concentrations of uranium, thorium and potassium in the silica. Samples were drawn from each drum, blended and split and are assumed to be representative.

uncertainties reflect maximum cumulative errors due to scale readability. These potential errors represent negligible contributions to the concentration uncertainties in RGTh-1.

Each of the two lots were blended for 16 h at a rotation rate of 30 revolutions per minute. Following each blending, the material was removed and stored in clean plastic pails. Approximately one half of each lot was combined and returned to the blender, blended for four hours and removed. The second half of each lot was then combined and blended for 16 h. This splitting and reblending process was repeated twice more to yield a total of six splits and 82 hours of blending in the production of RGTh-1.

Immediately following the final blending, RGTh-1 was bottled in nominal 500-g units using an auger-feed bottle filler. The bottle sequence was recorded for subsequent sampling for homogeneity testing. The yield was 1012 bottles. A screen-size analysis of a sample of RGTh-1 is shown in Table 6.

HOMOGENEITY OF RGTh-1

One bottle from each twenty-one bottle group in the bottling sequence was selected at random for homogeneity test measurements. Two samples of 225.0 g were drawn from each of the forty-nine test bottles and were packed uniformly to a density of 1.061 g/cm^3 in ten cm diameter thin steel cans. The samples remained sealed for five days prior to gamma-ray measurements.

The test measurements were gamma-ray emission intensity measurements of each sample in a randomized measurement sequence. In addition, sample A from bottle number 130 was re-measured following every sixth sample to provide an estimate of the measurement precision.

Emission counts in the ranges 0.4 to 3.40 (total), 2.42 - 3.40 (thorium window), 1.66 to 1.86 (uranium window) and 1.36 to 1.56 (potassium window) MeV were acquired for 60 s measurement periods(7). The measurements were made with the dual 5 x 5 inch NaI(Tl) spectrometer of the Airborne Geophysics Section, Geological Survey of Canada. The count data are compiled in Table 7, where A and B refer to the two samples from each bottle.

The distribution of sample counts about the mean values for each range are shown in Figures 1 - 4. Qualitatively, there are no observable trends with bottle number and the ranges of the distributions between samples are similar to those observed for the ranges of the repeatedly measured sample. Analysis of variance, one-way classification, fails to show any significance of the "between-bottles" component relative to the "within-bottle" component for each gamma-ray range, as shown in Tables 8 to 11(5). Physically, we conclude that there is no statistical evidence from these tests for an inhomogeneous distribution of thorium, uranium or potassium concentrations between bottles of RGTh-1.

Similarly, the statistical hypothesis that the between sample variance is equal to the measurement variance is tested by comparison of the ratio of the overall mean square estimate between samples to that obtained from the repeated sample measurement (s^2_s/s^2_m) to the critical value of the F statistic (Table 12). For each gamma-ray range, we conclude that there is no statistical evidence that the between samples variance is larger than the measurement variance at the 5% significance level of the test. Physically, we conclude that any residual component of variance due to concentration variance between samples, i.e. inhomogeneity, was not detected.

CONFIRMATION OF MASS FRACTION OF OKA-2 IN RGTh-1

The objective that RGTh-1 contain 800 µg/g thorium required that OKA-2 comprise 2.765 percent of the matrix. To confirm that the preparative dilution had been carried out accurately, six small samples were prepared using independent laboratory balances, for comparison to samples of RGTh-1. For each, 6.3580 ± 0.0002 g of OKA-2 and 223.64 ± 0.01 g of $Si0_2$ were combined and thoroughly blended. Samples of 225.00 ± 0.01 g were then sealed in steel cans for gamma-ray measurements of thorium to be compared with samples of RGTh-1 prepared identically (for the homogeneity measurements).

The measurement data are compiled in Table 13. The six laboratory samples and one canned sample of RGTh-1 were counted alternately for 60 s in a single measurement session as described for the homogeneity test series. Counts per minute for the silica employed in each preparation and water

(ambient background) based on 1800 measurements are included for comparison. The ratios (RGTh-1)/(Laboratory preparation) of total, thorium window; uranium window and potassium window net cmp are 0.996 ± 0.007, 0.994 ± 0.051, 1.04 ± 0.07 and 1.01 ± 0.04, respectively, where the uncertainties were propagated at the 95% confidence level. It is inferred that the large scale dilution was performed accurately.

RECOMMENDED VALUES FOR THORIUM, URANIUM AND POTASSIUM IN RGTh-1 AND CONCLUSION

From the known concentrations of thorium, uranium and potassium of the silica and OKA-2 components of RGTh-1 and the known amounts of each component in the blended material, the recommended concentrations for RGTh-1 are 800.2 ± 15.8 µg/g thorium, 6.26 ± 0.42 µg/g uranium and 0.02 ± 0.01 percent potassium, where the uncertainties represent 95% confidence interval estimates. The thorium/uranium concentration ratio is calculated as 127.8 ± 8.8. The compositions and uncertainty estimates of RGTh-1 and its components are summarized in Table 14.

The contribution of the silica component to the thorium concentration of RGTh-1 is negligible. That is, if the thorium concentration of the silica was known to be zero with absolute certainty, the recommended value would be calculated as 799.9 ± 15.8 µg/g. The recommended thorium concentration therefore derives virtually entirely from the interlaboratory certification and the accurately measured dilution ratio. Since the homogeneity of thorium in RGTh-1 has been demonstrated, the thorium concentration is regarded as meeting the criteria for certification of CCRMP.

Similarly, the contribution of the silica component to the concentration of uranium (and its radioactive daughters) is small. If the uranium content of the silica was absolutely zero, we would obtain 6.04 ± 0.22 µg/g for the recommended value and its estimated 95% confidence interval. Thus the silica contributes about 3.5% of the equivalent uranium content of RGTh-1 and doubles the uncertainty range. The recommended useage of the uranium value is the application of minor corrections for uranium interference on the radiometric response for thorium of RGTh-1. In view of

the high thorium/uranium ratio of RGTh-1, however, any such interference is likely to be small. From the concentrations and specific activities, the activity ratio $^{232}Th/^{238}U$ for RGTh-1 is calculated as 41.9 ± 2.9 (8).

The potassium content of the silica (0.01 ± 0.01%) contributes significantly to the potassium concentration of RGTh-1. If the potassium content were zero, the recommended value for RGTh-1 would be 0.0095 ± 0.0006 %. The measured value for silica thus doubles both the potassium concentration and uncertainty estimates for RGTh-1. From the concentrations and specific activities of thorium-232 and potassium-40, the activity ratio $^{232}Th/^{40}K$ is 570 ± 300 (8). Consequently, potassium may be regarded as a non-interferant for radiometric applications of RGTh-1.

In conclusion, it may be stated that RGTh-1 is an acceptable reference material for the radiometric determination of thorium in geological materials. It is particularly useful as a gamma-ray spectrometry calibration reference material, since interferences due to uranium and potassium are expected to be negligible for most applications. In conjunction with the use of the complementary materials RGU-1 and RGK-1, accurate calibration for the radioactive geological analytes should be greatly facilitated.

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Mr. P.B. Holman of the Airborne Geophysics Section, Geological Survey of Canada performed the homogeneity test measurements.

The authors also acknowledge the major contributions of the participating laboratories in the certification programs for OKA-2 who are listed in the Appendix.

The members of the IAEA consultants' committee and A.Y. Smith of IAEA initiated and steered this project to completion.

Dr. R. Sutarno of the Extraction Metallurgy Laboratory, CANMET reviewed this report and made substantial contributions to the discussion of statistical features.

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APPENDIX

PARTICIPANTS IN THE ANALYTICAL MEASUREMENT PROGRAMS FOR OKA-2

Participants in the Analytical Measurement Programs for OKA-2

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* Contributors via the IAEA measurement project.

Table 1 - Results of statistical assessments of thorium concentrations in OKA-2

	Data Base		
	IAEA	CCRMP	COMBINED
Recommended value, Th %	2.898	2.917	2.893
Confidence interval, Th %	± 0.072	± 0.077	± 0.057
No. accepted sets	15	17	29
No. accepted results	80	84	155
No. outlier sets	1	2	2

Table 2 - Results of statistical assessment of uranium concentrations in OKA-2

	Data Base			
	IAEA	CCRMP	Radium (as eU)	Combined
Recommended value, U µg/g	217.6	216.5	211.9	218.6
Confidence interval, U µg/g	± 31.4	± 12.2	± 21.7	± 8.1
No. accepted sets	12	13	7	27
No. accepted results	68	65	38	145
No outlier sets	0	1	2	6

Table 3 - Results of statistical assessment of potassium concentration in OKA-2

Recommended value, K %	0.344
Confidence interval K %	± 0.021
No. accepted sets	8
No. accepted observations	45
No. outlier sets	2

Table 4 - Approximate composition of OKA-2

Component	Wt. %
$RE_2O_3+Y_2O_3$	33.5 (±1)
CaO	25.3
SiO_2	14.7
P_2O_5	8.9
Fe_2O_3	5.8
ThO_2	3.29
F	1.3
MgO	1.2
Al_2O_3	0.8
SrO	0.7
MnO	0.5
ZnO	0.5
NiO	0.2
TiO_2	0.6
Na_2O	0.4
K_2O	0.41
C	0.3
U_3O_8	0.0258

Table 5 - Results of silica analysis by gamma-ray spectrometry

Drum	Th, µg/g		U, µg/g		K, %	
	CANMET	GSC	CANMET	GSC	CANMET	GSC
1	0.33	0.04	0.15	0.25	0.004	0.01
	0.31		0.17		0.003	
	0.41		0.20		0.006	
2	0.33	0.66	0.24	0.19	0.004	0.00
	0.34		0.21		0.001	
3	0.43	0.70	0.20	0.30	0.011	0.01
4	0.43	0.01	0.18	0.23	0.008	0.04
	0.45		0.18		0.009	
5	0.40	0.45	0.22	0.20	0.009	0.02
6	0.37	0.30	0.18	0.29	0.007	0.00
mean	0.38	0.36	0.19	0.24	0.006*	0.013
s.d.	0.05	0.30	0.03	0.05	0.003	0.015

* Calibration factor uncertainty is 143%(2σ)

Table 6 - Wet screen analysis of RGTh-1

Particle Size (µm)	Wt %
150 to 75	2.3
74 to 45	8.9
44 to 38	10.9
less than 38	77.9

Table 7 - IAEA RM RGTH-1 - Homogeneity Measurements by Gamma-Ray - Nov. 1986

Bot	Gam A	Gam B	Th A	Th B	K A	K B	U A	U B
6	18360.	18705.	1697.	1832.	759.	723.	397.	392.
40	18947.	18419.	1839.	1785.	806.	792.	401.	398.
54	18740.	18435.	1826.	1788.	792.	775.	411.	404.
92	18596.	18898.	1767.	1820.	750.	778.	417.	409.
96	18493.	18738.	1726.	1789.	758.	807.	406.	436.
124	18572.	18748.	1782.	1822.	750.	692.	376.	417.
130	18442.	18542.	1770.	1769.	750.	748.	426.	394.
155	18350.	18745.	1776.	1866.	742.	758.	392.	412.
179	18568.	18852.	1702.	1698.	737.	738.	406.	426.
198	18742.	18824.	1752.	1888.	793.	789.	434.	393.
229	18636.	18480.	1831.	1758.	748.	712.	398.	416.
237	18526.	18692.	1745.	1860.	752.	737.	395.	438.
264	18571.	18614.	1711.	1787.	810.	773.	369.	432.
278	18529.	18663.	1786.	1826.	765.	737.	380.	384.
300	18773.	18842.	1813.	1833.	740.	802.	404.	404.
319	18762.	18655.	1882.	1825.	729.	745.	421.	359.
338	18494.	18755.	1779.	1698.	807.	788.	381.	432.
376	18615.	18481.	1765.	1812.	798.	782.	378.	391.
384	18530.	18824.	1771.	1841.	780.	797.	386.	416.
416	18533.	18566.	1768.	1725.	755.	731.	413.	399.
427	18654.	18876.	1787.	1808.	756.	758.	423.	387.
454	18415.	18714.	1760.	1731.	751.	755.	412.	457.
472	18658.	18586.	1798.	1759.	820.	776.	362.	418.
486	18711.	18850.	1733.	1812.	740.	769.	414.	392.
512	18751.	18561.	1802.	1838.	713.	779.	423.	387.
531	18488.	18486.	1741.	1768.	740.	767.	435.	397.
558	18764.	18659.	1834.	1792.	756.	725.	447.	408.
572	18744.	18583.	1752.	1780.	768.	749.	433.	422.
609	18596.	18837.	1706.	1796.	735.	746.	400.	437.
613	18845.	18524.	1853.	1803.	761.	717.	436.	396.
639	18680.	18471.	1775.	1708.	781.	749.	440.	419.
672	18434.	18700.	1749.	1748.	742.	785.	392.	426.
681	18549.	18580.	1751.	1769.	786.	774.	393.	402.
712	18750.	18528.	1785.	1852.	807.	750.	430.	382.
734	18820.	18658.	1810.	1829.	773.	687.	377.	400.
753	18778.	18650.	1754.	1753.	764.	708.	431.	408.
770	18678.	18707.	1839.	1760.	787.	709.	408.	420.
782	18528.	18650.	1852.	1767.	750.	764.	382.	420.
806	18789.	18621.	1730.	1732.	824.	738.	427.	403.
838	18500.	18375.	1756.	1747.	739.	746.	403.	421.
845	18366.	18727.	1751.	1732.	735.	752.	391.	439.
879	18694.	18581.	1728.	1785.	808.	746.	427.	397.
899	18779.	18358.	1777.	1768.	776.	758.	413.	420.
905	18435.	18199.	1793.	1749.	734.	724.	384.	367.
940	18590.	18559.	1798.	1732.	776.	703.	393.	425.
949	18577.	18377.	1769.	1745.	783.	727.	419.	408.
972	18582.	18648.	1767.	1841.	791.	760.	393.	418.
989	18432.	18646.	1797.	1752.	741.	786.	394.	434.
1012	18696.	18684.	1808.	1749.	734.	820.	412.	392.
130	18442.		1770.		750.		426.	
130	18636.		1735.		767.		389.	
130	18587.		1716.		781.		407.	
130	18615.		1793.		749.		434.	
130	18780.		1764.		792.		423.	
130	18497.		1791.		740.		388.	
130	18660.		1788.		774.		406.	
130	18900.		1760.		760.		423.	
130	19656.		1891.		693.		402.	
130	18537.		1736.		797.		428.	
130	18727.		1766.		774.		408.	
130	18458.		1717.		741.		399.	
130	18716.		1749.		773.		410.	
130	18607.		1824.		744.		402.	
130	18535.		1794.		741.		393.	
130	18093.		1754.		697.		400.	
130	18927.		1848.		778.		396.	

Table 8 - Analysis of variance of total gamma-ray counts for between- and within-bottle components

Source of Variation	D.F.	Sum of Squares	Mean Squares	F-Ratio	F-Table
Between sets	48	.90526467E+06	.18859681E+05	.796	1.610
Within sets	49	.11606115E+07	.23685949E+05		
Total	97	.20658762E+07			

Table 9 - Analysis of variance of thorium-window gamma-ray counts for between- and within-bottle components

Source of Variation	D.F.	Sum of Squares	Mean Squares	F-Ratio	F-Table
Between sets	48	.10406400E+06	.21680000E+04	1.231	1.610
Within sets	49	.86324000E+05	.17617143E+04		
Total	97	.19038800E+06			

Table 10 - Analysis of variance of uranium-window gamma-ray counts for between- and within-bottle components.

Source of Variation	D.F.	Sum of Squares	Mean Squares	F-Ratio	F-Table
Between sets	48	.14137837E+05	.29453827E+03	.586	1.610
Within sets	49	.24616500E+05	.50237755E+03		
Total	97	.38754337E+05			

Table 11 - Analysis of variance of potassium-window gamma-ray counts for between- and within bottle components

Source of Variation	D.F.	Sum of Squares	Mean Squares	F-Ratio	F-Table
Between sets	48	.42216347E+05	.87950723E+03	1.038	1.610
Within sets	49	.41525500E+05	.84745918E+03		
Total	97	.83741847E+05			

Table 12 - Comparison of between-samples and measurement (within-sample) mean square estimates of variances of gamma-ray counts

Test	Mean Squares		Ratio	$F_{\alpha=0.05}$ (97,16)
	Between Samples (97 d.f.)	Measurement (16 d.f.)		
Total gamma	2.130 E4	3.642 E4	0.583	2.08
Th Window	1.963 E3	2.096 E3	0.936	
K Window	8.633 E2	8.466 E2	1.020	
U Window	3.995 E2	2.004 E2	1.994	

Table 13 - Confirmation of accuracy of dilution ratio of OKA-2/SiO_2 in the preparation of RGTh-1

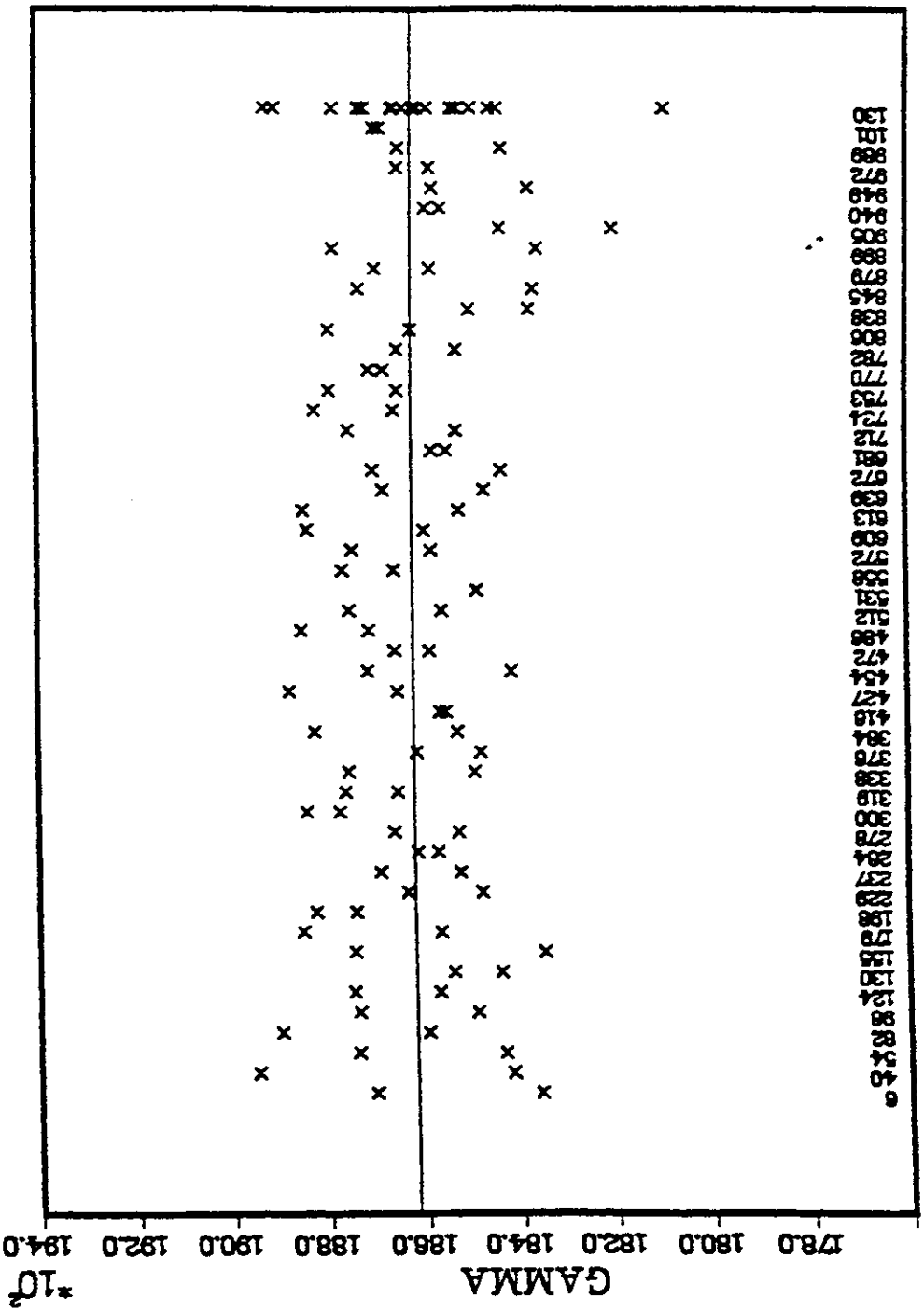
	Gamma Ray Counts Per Minute							
	Total (0.4 - 3.4 MeV)		Th Window (2.42 - 3.40 MeV)		U Window (1.66 - 1.86 MeV)		K Window (1.36 - 1.56 MeV)	
	RGTh-1	OKA-2/SiO_2	RGTh-1	OKA-2/SiO_2	RGTh-1	OKA-2/SiO_2	RGTh-1	OKA-2/SiO_2
1	18580	18526	1858	1715	416	400	744	735
2	18349	18597	1793	1781	419	410	753	723
3	18574	18504	1746	1759	397	423	727	707
4	18467	18543	1723	1917	419	399	711	754
5	18536	18706	1776	1816	393	397	744	740
6	18404	18471	1775	1741	457	385	747	715
Mean*	18485 ± 100	18558 ± 88	1778.5 ± 49	1788.2 ± 75.5	416.8 ± 23.9	402.3 ± 13.5	737.7 ± 15.7	727.0 ± 18
SiO_2**	571 ± 11	522 ± 10	37.0 ± 2.7	34.6 ± 2.5	26.8 ± 2.3	25.8 ± 2.3	54.5 ± 3.3	53.2 ± 3.3
H_2O**	530 ± 11	530 ± 11	34.5 ± 2.6	34.5 ± 2.6	22.3 ± 2.1	22.3 ± 2.1	50.6 ± 3.2	50.6 ± 3.3

* error estimates are 95% confidence interval for mean
** error estimates are 2σ from counting statistics

Table 14 - Composition of RGTh-1 and its components

	OKA-2	Silica	RGTh-1
kg	14.513 ± 0.004	510.4 ± 0.6	524.9 ± 0.6
Th (µg/g)	28930 ± 570	0.37 ± 0.05	800.2 ± 15.8
U (µg/g)	218.6 ± 8.1	0.22 ± 0.05	6.26 ± 0.42
K (%)	0.344 ± 0.021	0.01 ± 0.01	0.019 ± 0.010

Figure 1


IAEA RM RGTH-1 — HOMOGENEITY MEASUREMENTS BY GAMMA-RAY — NOV. 1986
GAMMA
*10²
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180.0
182.0
184.0
186.0
188.0
190.0
192.0
194.0
BOTTLE NO.

Figure 2

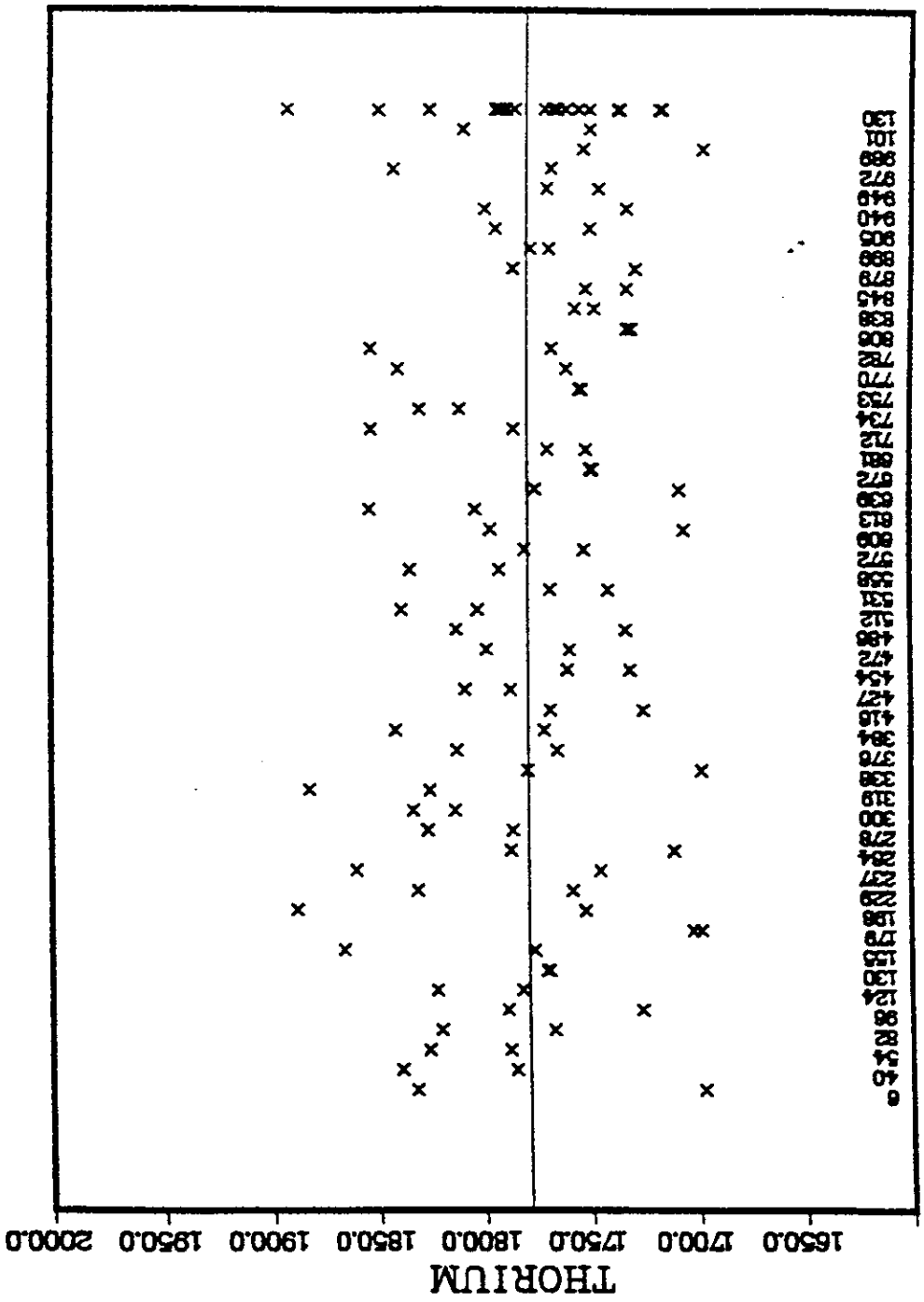

IAEA RM ROTH-1 —— HOMOGENEITY MEASUREMENTS BY GAMMA-RAY —— NOV. 1986
THORIUM
1650.0
1700.0
1750.0
1800.0
1850.0
1900.0
1950.0
2000.0
BOTTLE NO.

Figure 3

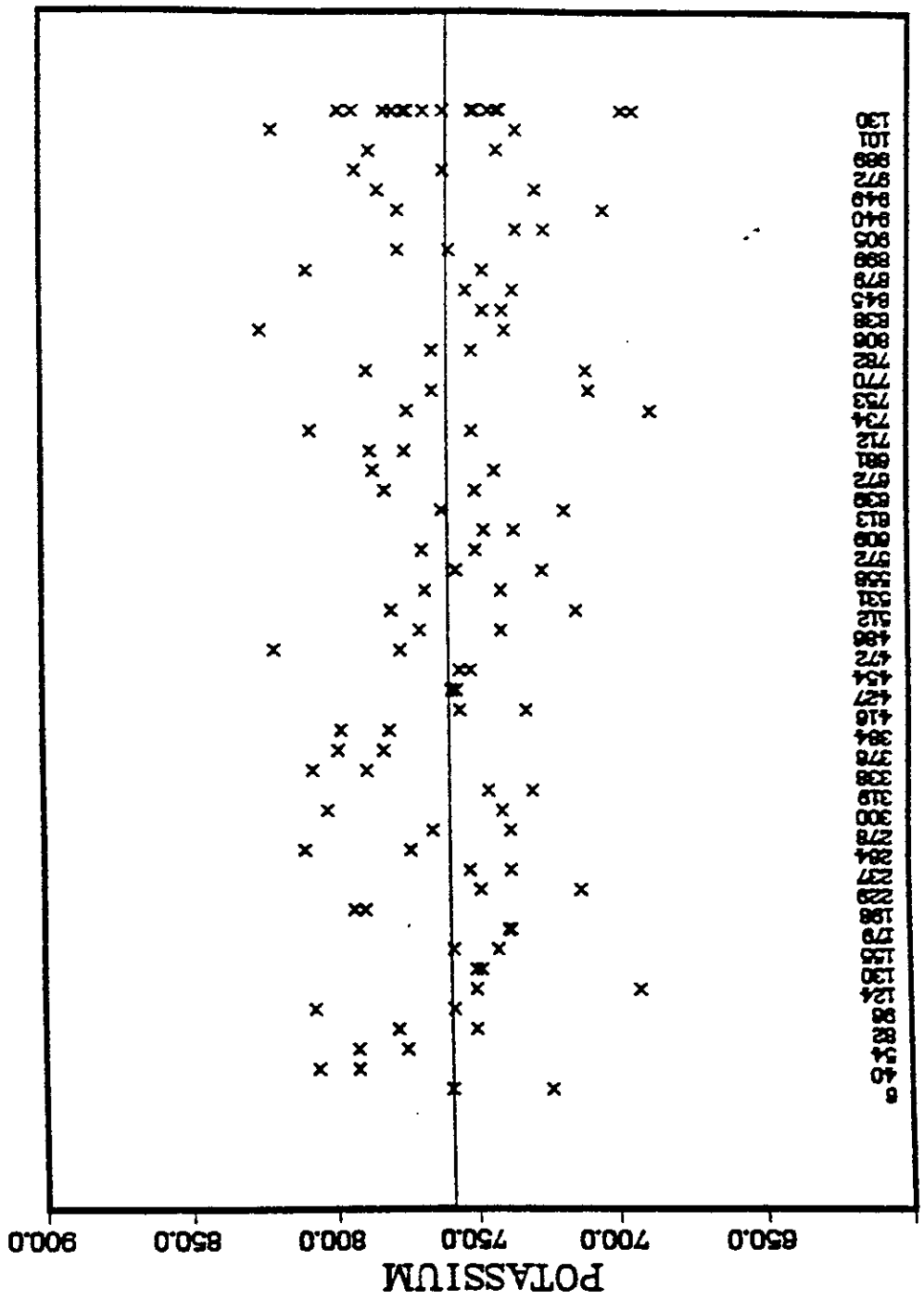
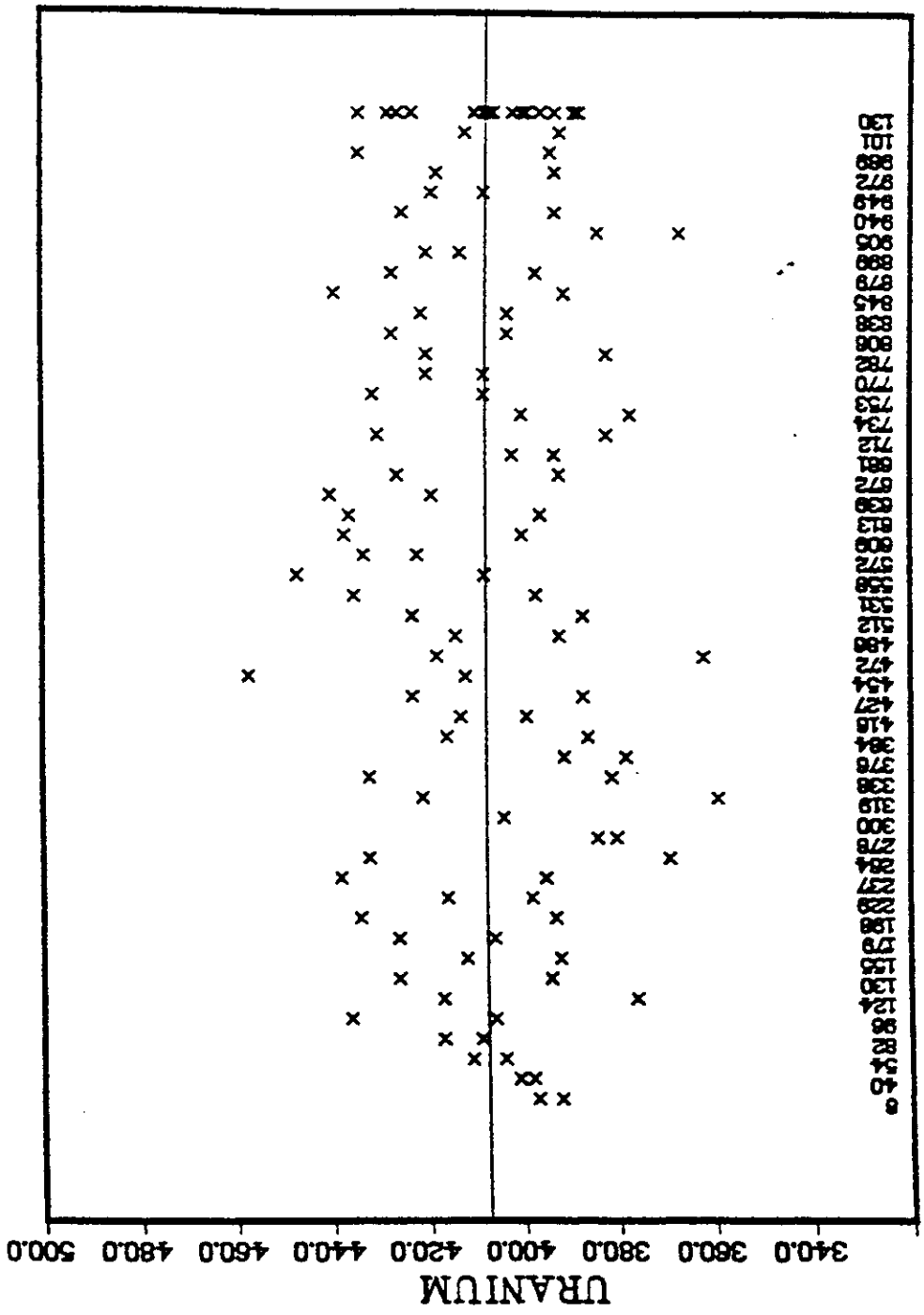

IAEA RM RGTH-1 —— HOMOGENEITY MEASUREMENTS BY GAMMA-RAY —— NOV. 1986
POTASSIUM
900.0
850.0
800.0
750.0
700.0
650.0
BOTTLE NO.

Figure 4


IAEA RM RGTH-1 —— HOMOGENEITY MEASUREMENTS BY GAMMA-RAY —— NOV. 1986
URANIUM
340.0
360.0
380.0
400.0
420.0
440.0
460.0
480.0
500.0
BOTTLE NO.

PREPARATION AND CERTIFICATION OF RGK-1 A POTASSIUM RADIOMETRIC REFERENCE MATERIAL

by

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Description of the material

A batch of 500 kg of potassium sulphate was supplied by Merck Company as Potassium sulphate powder Extra Pure DAC (Merck Catalogue No. 5151). The results of analysis provided by the supplier were as follows:

1.	Potassium sulphate (acidimetric)	99.8%
2.	Chloride (Cl')	< 5 µg/g
3.	Total nitrogen	< 8 µg/g
4.	Heavy metals (as Pb)	< 10 µg/g
5.	Compounds to be reduced by potassium permanganate (as SO_3)	< 40 µg/g
6.	Arsenic	< 2 µg/g
7.	Lead	< 10 µg/g
8.	Calcium	< 20 µg/g
9.	Iron	< 10 µg/g
10.	Copper	< 10 µg/g
11.	Magnesium	< 20 µg/g
12.	Sodium	< 50 µg/g
13.	Zinc	< 25 µg/g
14.	Loss on ignition (600° C)	< 0.1%
15.	Water	< 0.1%

The homogeneity of potassium in the material was checked by atomic absorption spectrometry in twenty samples taken at random from the batch. It was found that the material may be considered homogeneous for potassium, at least for a sample weight equal to or larger than 0.1 g.

The results of sieve analysis were as follows:

Grain size (mm)	wt. %
0.20 - 0.16	8
0.16 - 0.10	31
less than 0.10	61

Aliquots of 500 g of the material were transferred to plastic bottles, labeled IAEA/RGK-1.

Establishing the Certified Values

The content of potassium sulphate certified by the supplier, 99.8 % K_2SO_4 (44.8 % K) was determined as sulphate. The concentration of potassium was determined directly as potassium by atomic absorption spectrometry in the Agency's Laboratories, Chemistry Unit. In order to achieve good precision and accuracy of the results, twenty samples of the material were analyzed. The average of these determinations was 45.0 % of potassium with an uncertainty of ±0.3 % at a significance level of 0.05. This value agrees with the value provided by the supplier thus the value of the supplier was accepted as the recommended value for the potassium concentration with ± 0.3% confidence interval.

The concentrations of uranium and thorium were estimated by the Agency's Laboratories, Chemistry Unit using fluorimetry and neutron activation analysis, respectively.

INTERNATIONAL ATOMIC ENERGY AGENCY

REFERENCE MATERIAL FOR GAMMA-RAY SPECTROMETRIC ANALYSIS OF GEOLOGICAL MATERIALS

IAEA/RGU-1

CERTIFICATE OF ANALYSIS

COMPONENT	CONCENTRATION*	CONFIDENCE INTERVAL**
Uranium	400 µg/g	± 2 µg/g
Thorium	less than 1 µg/g	---
Potassium	less than 20 µg/g	---

*Expressed on dry weight basis (constant weight at 130°C)
**At a significance level of 0.05

DESCRIPTION OF MATERIAL

RGU-1, RGTh-1 and RGK-1 are intended for use in calibrating laboratory gamma-ray spectrometers for the determination of U, Th and K in geological materials. RGU-1 was prepared by the Canada Centre for Mineral and Energy Technology (CANMET) under a contract with the International Atomic Energy Agency. The material was prepared by dilution of Canada Certified Reference Material Project (CCRMP) uranium ore BL-5 (7.09% U) with a floated silica powder of similar grain size distribution. BL-5 has been certified for uranium, ^{226}Ra and ^{210}Pb confirming that it is in radioactive equilibrium. The complete description of the preparation and certification of RGU-1 may be found in the reference.

REFERENCE

Preparation of Gamma-ray Spectrometry Reference Materials RGU-1, RGTh-1 and RGK-1 Report - IAEA/RL/148, Vienna, 1987

This report may be obtained from:
INTERNATIONAL ATOMIC ENERGY AGENCY
Agency's Laboratories
Analytical Quality Control Services
P.O.Box 100
A-1400 Vienna, AUSTRIA

INTERNATIONAL ATOMIC ENERGY AGENCY

REFERENCE MATERIAL
FOR
GAMMA-RAY SPECTROMETRIC ANALYSIS
OF
GEOLOGICAL MATERIALS

IAEA/RGTh-1

CERTIFICATE OF ANALYSIS

COMPONENT	CONCENTRATION*	CONFIDENCE INTERVAL**
Thorium	800 µg/g	± 16 µg/g
Uranium	6.3 µg/g	± 0.4 µg/g
Potassium	0.02%	± 0.01%

*Expressed on dry weight basis (constant weight at 130°C)
**At a significance level of 0.05

DESCRIPTION OF MATERIAL

RGU-1, RGTh-1 and RGK-1 are intended for use in calibrating laboratory gamma-ray spectrometers for the determination of U, Th and K in geological materials. RGTh-1 was prepared by the Canada Centre for Mineral and Energy Technology (CANMET) under a contract with the International Atomic Energy Agency. The material was prepared by dilution of Canada Certified Reference Material Project (CCRMP) thorium ore OKA-2 (2.89% Th, 219 µg/g U) with a floated silica powder of similar grain size distribution. The agreement between radiometric and chemical measurements of thorium and uranium in OKA-2 shows both series to be in radioactive equilibrium. The complete description of the preparation and certification of RGTh-1 may be found in the reference.

REFERENCE

Preparation of Gamma-ray Spectrometry Reference Materials RGU-1, RGTh-1 and RGK-1 Report - IAEA/RL/148, Vienna, 1987

This report may be obtained from:
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INTERNATIONAL ATOMIC ENERGY AGENCY

REFERENCE MATERIAL
FOR
GAMMA-RAY SPECTROMETRIC ANALYSIS
OF
GEOLOGICAL MATERIALS

IAEA/RGK-1

CERTIFICATE OF ANALYSIS

COMPONENT	CONCENTRATION*	CONFIDENCE INTERVAL**
Potassium	44.8 %	± 0.3 %
Uranium	less than 0.001 µg/g	
Thorium	less than 0.01 µg/g	

*Expressed on dry-weight basis (constant weight at 130°C)
**At a significance level of 0.05

DESCRIPTION OF MATERIAL

RGU-1, RGTh-1 and RGK-1 are intended for use in calibrating laboratory gamma-ray spectrometers for the determination of U, Th and K in geological materials. RGK-1 is intended for use in calibrating laboratory gamma-ray spectrometers for the determination of U, Th and K in geological materials. The material is extra pure (99.8%) potassium sulphate supplied by Merck Company. The potassium value and its uncertainty were obtained from repeated measurements by atomic absorption spectrometry in the IAEA Laboratory which confirmed the potassium sulphate value certified by Merck. The upper limits of the uranium and thorium values were estimated by the IAEA Laboratory using fluorimetry and activation analysis, respectively. A complete description of RGK-1 may be found in the reference.

REFERENCE

Preparation of Gamma-ray Spectrometry Reference Materials RGU-1, RGTh-1 and RGK-1 Report - IAEA/RL/148, Vienna, 1987

This report may be obtained from:
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