

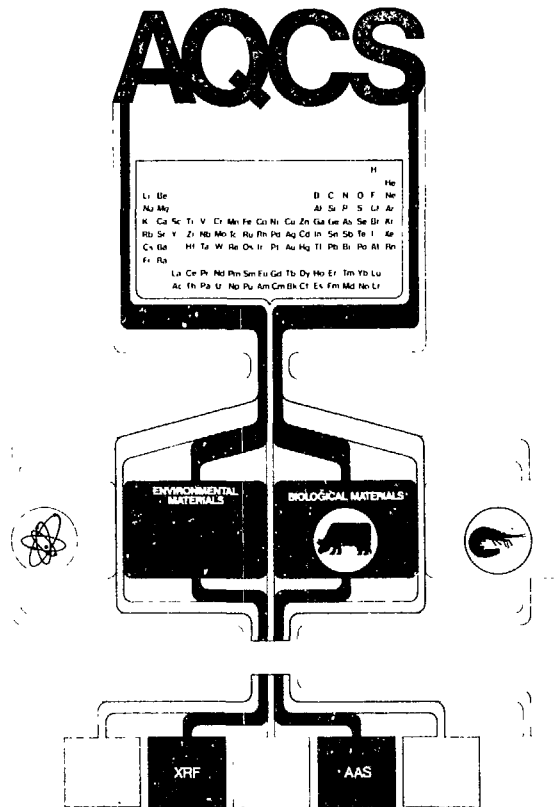
REPORT ON THE
INTERCOMPARISON RUN

IAEA-314

Ra-226, Th and U

IN

STREAM SEDIMENT



ANALYTICAL QUALITY CONTROL SERVICES

INTERNATIONAL ATOMIC ENERGY AGENCY P.O. BOX 100, A 1400 VIENNA, AUSTRIA



IAEA/AL/038
February 1991

Intercomparison Study IAEA - 314
on the Determination of U, Th and Ra-226 in Stream Sediment

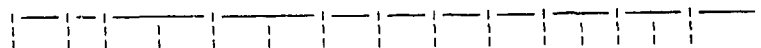
by

V. Strachnov, V. Valkovic, R. Zeisler, R. Dekner

International Atomic Energy Agency
Agency's Laboratories
Analytical Quality Control Services

Vienna, February 1991

WP1723k



Summary

This report contains the results of the intercomparison IAEA - 314 on the determination of uranium, thorium and Ra-226 in stream sediment.

The participants included 39 laboratories located in 19 countries, and statistical evaluation of their data yield recommended values for these elements.

The elements, their recommended values and confidence intervals are listed below:

Ra - 226	732 Bq/kg (678-787)
Th	17.8 microg/g (16.8-18.8)
U	56.8 microg/g (52.9-60.7)

Reference date: 30 January, 1988

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1. Introduction

In 1990, the Agency's Laboratory in Seibersdorf distributed two new stream sediment samples with quantities of some artificial and natural radionuclides and collected from different places. It was anticipated that the concentrations of the analytes in these sediments would be different and, therefore, would constitute excellent intercomparison samples, at the same time, knowledge of the concentration of specific analytes would provide essential for understanding of the development of sediments from water bodies on a broad geographical scale.

One was a sediment from the Sibolga area on the West coast of north Sumatra, Indonesia. The another sample was collected from the Kalan area of the West Kalimantan (Borneo), Indonesia. The result of the previous inter-comparison has been published in IAEA/AL/037

2. Scope of Study

Participating laboratories were requested to determine Radium-226, Uranium and Thorium. The analysts were requested to make at least three, but preferably six independent measurements for each element.

3. Description of the material

The bulk stream sediment sample donated by the Indonesian Atomic Energy Commission (IAEC) was collected from the Kalan area of the West Kalimantan (Borneo), Indonesia.

The sun dried batch of the stream sediment passing a 180 μm sieve was further ground and homogenized.

Aliquots of 50g were distributed in plastic bottles and all bottles were sealed and sterilized by gamma ray irradiation to a total dose of $2.5 \cdot 10^4$ Gy using a ^{60}Co source.

The homogeneity of the material (after bottling) was checked by determining the concentration of uranium by laser fluorimetry in two sub-samples taken from various bottles chosen at random.

By applying the F-test it was found that the results did not differ significantly for uranium and that this material could be considered homogeneous for a sample size of greater or equal to 500 mg (significance level of 0.05).

4. Evaluation of results

The original data that were received from the participating laboratories were edited (converted to the same units and format) before being entered into a computer data file. This data file was processed by a computer program especially written for evaluating intercomparison results. Results which deviated significantly from the population were considered to be outliers and rejected if they failed either one of the following statistical tests at the significance level of $\alpha = 0.05$:

- 1) Dixon's
- 2) Grubb's
- 3) coefficient of skewness, and
- 4) coefficient of kurtosis.

Additional information on acceptance criteria can be found in reference (1).

4.1 Explanation of tables

4.1.1. Data tables

The laboratory mean values for a specific element for which at least two laboratory means were supplied are presented in Tables 1 to 3.

LAB. CODE NO.: Each laboratory was assigned a code number, which is the same throughout the report. To ensure anonymity these code numbers do not correspond to the sequence of the laboratories in the list of participants given at the end of the report.

METHOD CODE: The analytical techniques employed by the participating laboratories are represented in the form of codes (a letter and number). The key to the different analytical techniques is given in Table A

NO. OF DETERMINATIONS: The number of individual results for a given element reported by the participating laboratory.

LAB. MEAN: The arithmetic mean computed from all the individual results reported by the participating laboratory. An asterisk (*) after the lab mean denotes that it was detected and rejected as an outlier. Outliers were not used to compute the overall mean for this element.

LAB. STANDARD DEVIATION: The absolute and relative standard deviations were calculated if at least three results were reported by the participating laboratory.

[1] R. Dybczynski, A. Tugsavul, O. Suschny, Analyst 103 (1978), 733

4.1.2. Summary of results tables

The summary of the results for IAEA-314 is given in Table B. Most of the terms used in the summary table have been already defined. The standard error (S.E.) is defined as the standard deviation of the mean values divided by the square root of the number of laboratory means.

4.2. Description of figures

A figure was plotted for an element when at least five or more laboratory means were reported. The laboratory means are plotted in ascending order (microg/g or Bq/kg) on the y-axis with their corresponding laboratory code noted along the x-axis.

The reported uncertainties or "error bars" (standard deviation of the laboratory mean) are also shown. The code above the error bar is the laboratory method code, while the value below the error bar is the number of individual determinations. If a "less than" value was reported, an arrow downwards was employed rather than the numerical value and these values were not used to compute the overall mean. The mean value and the 95% confidence interval for all the accepted laboratory means are listed in the figure caption when appropriate. Finally, the solid points indicate those values that (filled in circles) were detected and rejected as outliers for the calculation of the overall laboratory mean.

4.3. Criteria for recommended values and confidence intervals.

Please note that these criteria are especially designed for this report and do not apply for general consideration.

The following criteria have been used for data classification:

1. The relative uncertainty of the overall mean (\bar{x}) at the significance level of $\alpha = 0.05$ defined as $\frac{SE \cdot t_{0.05}}{\bar{x}}$ is lower than a) 20%, b) 30%;
2. The overall mean was calculated on the basis of the results obtained from at least two different analytical methods.
3. The percentage of outliers was less than a) 20%, b) 30%;
4. At least 20 laboratory averages have been used for the calculation of the overall mean.

The overall mean for the particular element was classified as "reference value" (class A), when criteria 1a, 2, 3a, and 4 were fulfilled. When criteria 1b, 2, 3b, and 4 were fulfilled the overall mean of a given element was classified as "reference value" (Class B).

5. Results and conclusion

The results obtained from this intercomparison are detailed in Tables 1-4.

Since a large number of laboratories provided their results using a different analytical methods, it was possible to establish "recommended values" for uranium, thorium and Ra-226. A survey of outlying results seems to indicate that a relative small number of laboratories are mainly responsible for producing erroneous results, and not the analytical technique or the kind of element being determined. Certain differences between the methods, however, seem to exist in isolated cases and this problem deserves further investigation.

The recommended values for the activity concentration (Bq/kg) of Ra-226 and U, Th(microg/g) in IAEA-313 are summarized in Table C.

6. Acknowledgements

The authors of this report wish to thank to Ms. Katalin Lewis-Goettler for processing the results and to the Oesterreichisches Forschungszentrum for the ⁶⁰Co irradiation of these intercomparison samples. Last, we would also like to thank all the participating laboratories listed at the end of this report for their cooperation in providing the data for this intercomparison.

Table A

Laboratory method codes used in the IAEA - 314 Intercomparison

Method Code	Method
G2	Gamma ray spectrometry without sample pre-treatment
G3	Gamma ray spectrometry with sample pre-treatment
F1	Fluorimetry. Without specification
F2	Fluorimetry. Laser
K1	α - counting techniques
K2	α - spectrometry with specification
M1	Mass spectrometry without specification
M2	ICP - Mass spectrometry
N2	Neutron activation analysis. Thermal.Instrumental
N3	Neutron activation analysis. Epithermal.Instrumental
N7	Delayed neutron counting
R1	Radon emanation method
S2	Spectrophotometry without specification
T1	Fission track method
X1	X-ray fluorescence analysis

TABLE NO. 1 SIGNIFICANCE LEVEL 0.05

RESULTS OF INTERCOMPARISON FOR RA-226 IN STREAM SEDIMENT-314, 1990

UNIT: BQ/KG

NO.	LAB. CODE NO.	METHOD CODE	NO.OF DETERM.	MEAN	STANDARD DEV.		ACCEPTANCE CODE **
					ABS	REL %	
1	3	G2	6	766.67	30.77	4.0	OK
2	4	G2	4	700.00	9.83	1.4	OK
3	5	G2	5	817.00	3.39	0.4	8.7
4	6	R1	6	798.00	79.59	10.0	0.1
5	17	G2	6	544.88	7.99	1.5	-16.6
6	18A	R1	3	845.67	6.11	0.7	9.5
7	18B	R1	3	852.67	8.96	1.1	7.3
8	19	R1	4	509.25	18.83	3.7	-8.9
9	20	G2	2	1164.00*	7.66	0.5	66.6
10	21	G2	6	455.07	1.71	0.4	LO
11	22	R1	3	550.00	26.46	4.8	-4.8
12	23	G2	4	712.25	6.50	0.9	OK
13	24	R1	3	813.33	5.77	0.7	4.5
14	25	N2	6	551.33	18.08	3.3	-7.0
15	26	G2	6	850.67	20.71	2.4	3.1
16	27A	K1	1	980.00			HI
17	27B	G2	5	569.20	26.10	4.6	-4.2
18	28	R1	3	849.67	53.93	6.3	1.2
19	29	G2	6	711.00	24.12	3.4	OK
20	30	G2	6	736.17	49.90	6.8	OK
21	31	G2	1	840.00			HI
22	32	G2	6	719.65	8.68	1.2	OK
23	35A	G2	4	883.17	7.25	0.8	13.2
24	35B	G2	4	864.47	21.96	2.5	3.5
25	37	G2	3	814.57	1.36	0.2	20.0
26	38	G2	6	658.00	4.65	0.7	-4.2
27	39	G2	1	652.00			LO

** FOR CONFIDENCE INTERVAL 677.53 TO 787.45

RA-226 IN STREAM SEDIMENT-314, 1990 (BQ/KG)

MEAN VALUE: 732.49 BQ/KG

95 % CONFIDENCE INTERVAL: 677.53 TO 787.45

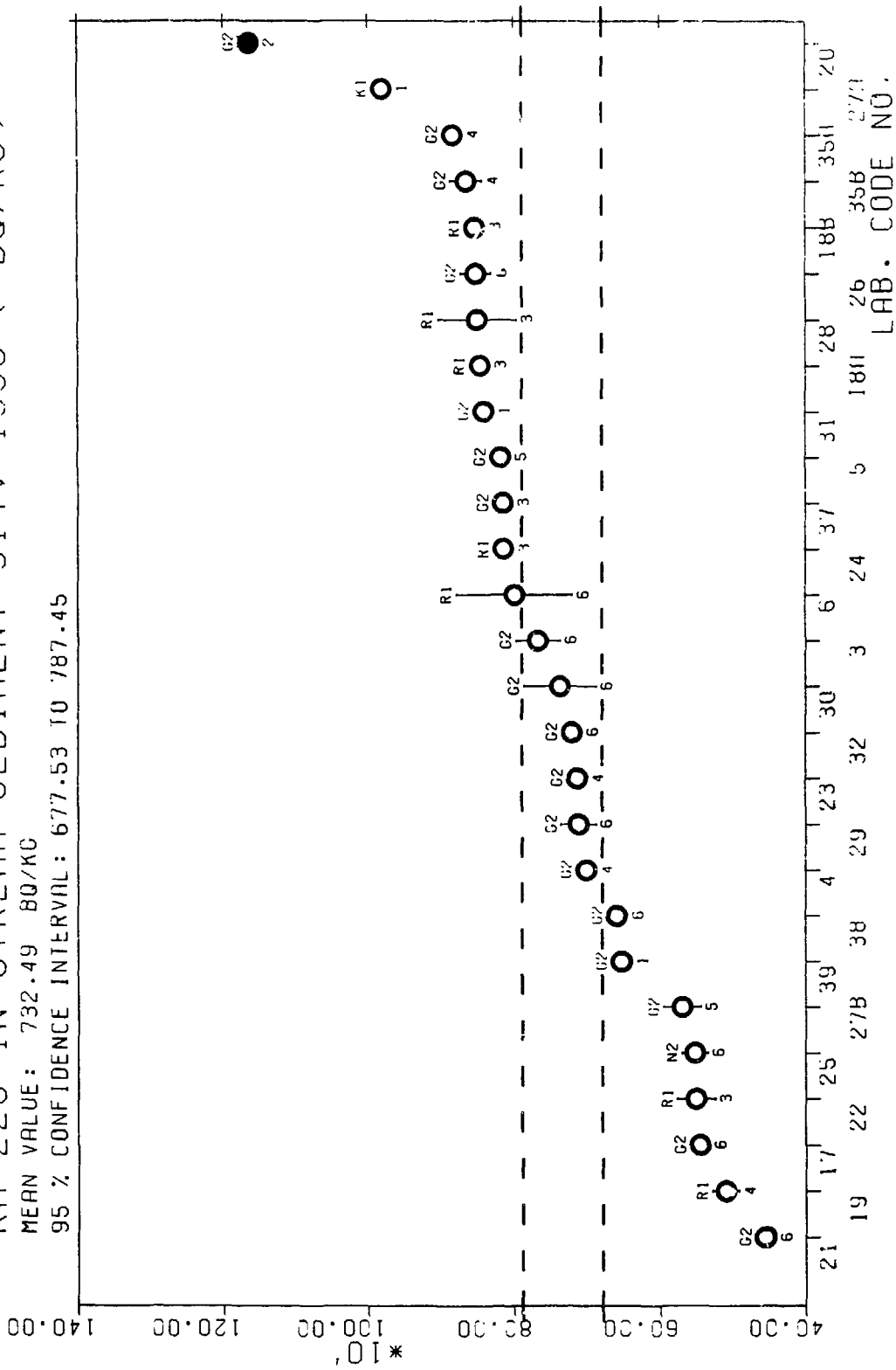


TABLE NO. 2 SIGNIFICANCE LEVEL 0.05

RESULTS OF INTERCOMPARISON FOR PH IN STREAM SEDIMENT-314, 1990

UNIT: MICRO/G

NO.	LAB. CODE NO.	METHOD CODE	NO.OF DETERM.	MEAN	STANDARD DEV.		ACCEPTANCE CODE **
					ABS	REL %	
1	1	X1	6	18.67	0.52	2.8	OK
2	2	G2	6	20.00	1.41	7.1	0.9
3	3	G2	6	19.17	1.17	6.1	0.3
4	4	G2	4	18.10	0.41	2.3	OK
5	7	N2	6	16.35	0.19	1.1	-2.2
6	9	X1	4	28.25*	1.89	6.7	5.0
7	10	X1	6	19.17	0.75	3.9	0.5
8	11	X1	4	6.70*	1.69	25.2	-6.0
9	12	N2	4	18.02	0.81	4.5	OK
10	14	S2	4	15.92	1.25	7.8	-0.7
11	15	N2	6	16.42	0.58	3.5	-0.6
12	16A	M2	6	13.43	0.29	2.1	-11.6
13	16B	N2	6	17.82	0.35	2.0	OK
14	17	G2	6	16.80	1.46	8.7	OK
15	18A	N2	6	18.70	0.41	2.2	OK
16	18B	N2	6	18.10	1.23	6.8	OK
17	18C	N2	6	31.53*	0.74	2.4	17.1
18	18D	N2	6	30.82*	1.65	5.4	7.3
19	19	N2	5	16.88	0.78	4.6	OK
20	21	G2	6	106.45*	4.51	4.2	19.4
21	22	K1	6	12.83	1.94	15.1	-2.0
22	24	G2	3	19.00	1.00	5.3	0.2
23	25	N2	6	11.88	0.70	5.9	-6.9
24	26	G2	6	19.20	1.37	7.2	0.3
25	27A	G2	5	21.72	3.28	15.1	0.9
26	27B	K1	2	20.55	0.78	3.8	2.3
27	29	G2	6	25.83*	3.37	13.1	2.1
28	30	N3	6	17.40	0.22	1.3	OK
29	32	N2	6	16.45	0.47	2.9	-0.7
30	35A	G2	4	20.97	0.79	3.8	2.8
31	35B	G2	4	20.75	1.74	8.4	1.1
32	36	N2	5	78.98*	3.90	4.9	15.4

** FOR CONFIDENCE INTERVAL 16.76 TO 18.79

TABLE NO. 3 SIGNIFICANCE LEVEL 0.05

RESULTS OF INTERCOMPARISON FOR TH-232 IN STREAM SEDIMENT-314, 1990

UNIT: MICRO/G

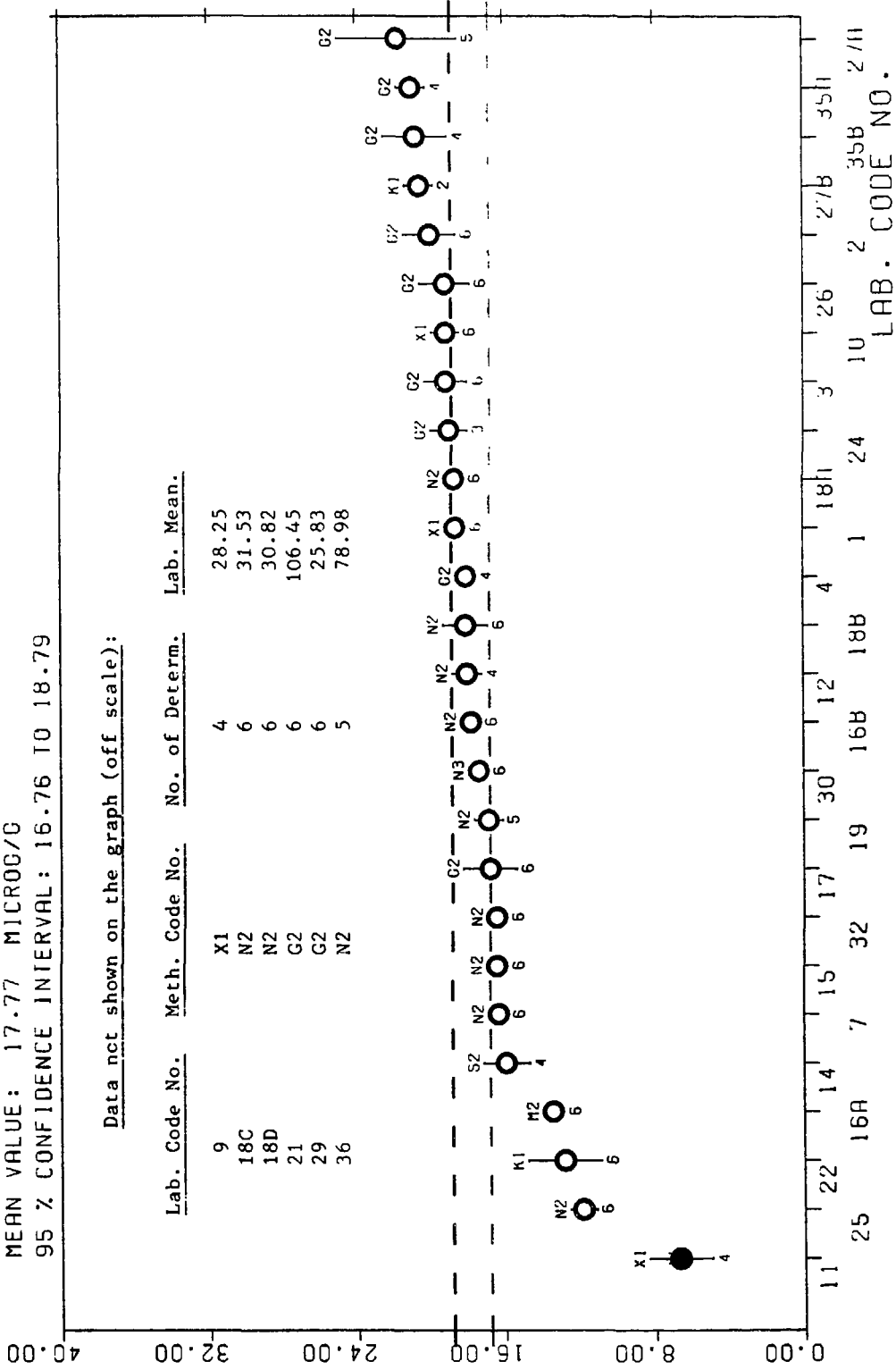
NO.	LAB. CODE NO.	METHOD CODE	NO.OF DETERM.	MEAN	STANDARD DEV.	
					ABS	REL %
1	37	G2	3	19.77	0.32	1.6

TH IN STREAM SEDIMENT-314, 1990 (MICROG/G)

MEAN VALUE: 17.77 MICROG/G
 95 % CONFIDENCE INTERVAL: 16.76 TO 18.79

Data not shown on the graph (off scale):

Lab. Code No.	Meth. Code No.	No. of Determ.	Lab. Mean.
9	X1	4	28.25
18C	N2	6	31.53
18D	N2	6	30.82
21	G2	6	106.45
29	G2	6	25.83
36	N2	5	78.98



LAB. CODE NO. 2/11 35B 2/11

TABLE NO. 4 SIGNIFICANCE LEVEL 0.05

RESULTS OF INTERCOMPARISON FOR U IN STREAM SEDIMENT-314, 1990

UNIT: MICROG/G

NO.	LAB. CODE NO.	METHOD CODE	NO.OF DETERM.	MEAN	STANDARD DEV.		ACCEPTANCE CODE **
					ABS	REL %	
1	1	X1	6	50.50	0.55	1.1	-4.3
2	2	G2	6	57.67	2.66	4.6	OK
3	3	G2	6	96.50*	1.52	1.6	23.6
4	4	G2	4	155.00*	4.55	2.9	20.7
5	6	F1	6	54.00	2.97	5.5	OK
6	7	N2	6	54.80	1.96	3.6	OK
7	8	F2	6	57.88	3.69	6.4	OK
8	9	X1	4	75.25	0.96	1.3	15.2
9	10	X1	6	106.67*	1.03	1.0	44.5
10	11	X1	4	25.87	2.19	8.5	-12.3
11	12	M1	3	58.83	0.72	1.2	OK
12	13	K2	3	149.63*	7.25	4.8	12.3
13	14	F1	4	44.57	2.21	5.0	-3.8
14	15	N2	6	47.17	1.66	3.5	-3.4
15	16A	M2	6	50.90	0.91	1.8	-2.2
16	16B	N7	6	53.63	0.57	1.1	OK
17	16C	N2	6	58.05	2.19	3.8	OK
18	17	G2	6	75.58	10.54	14.0	1.4
19	18A	N7	6	58.98	0.99	1.7	OK
20	18B	N7	6	58.73	1.06	1.8	OK
21	19	N2	5	52.28	0.79	1.5	-0.7
22	21	G2	6	82.35	1.98	2.4	10.9
23	22	F1	6	535.00*	33.91	6.3	14.0
24	24	G2	3	65.67	2.31	3.5	2.2
25	25A	N7	6	48.77	0.88	1.8	-4.7
26	25B	N2	6	36.22	2.75	7.6	-6.1
27	26	G2	6	58.92	5.25	8.9	OK
28	27A	G2	4	78.95	12.35	15.6	1.5
29	27B	K1	2	78.60	11.46	14.6	1.6
30	28	K2	3	58.30	1.20	2.1	OK
31	29	G2	6	47.00	1.26	2.7	-4.6
32	30	N3	6	55.90	0.36	0.6	OK
33	32	N2	6	52.55	1.58	3.0	-0.2
34	33A	T1	6	58.13	1.83	3.2	OK
35	33B	T1	6	52.88	3.16	6.0	OK
36	33C	T1	3	45.67	1.36	3.0	-5.3
37	34A	T1	6	60.70	4.54	7.5	0.0
38	34B	T1	6	57.27	1.97	3.4	OK
39	34C	T1	3	46.80	0.80	1.7	-7.6
40	35A	G2	4	62.97	1.78	2.8	1.3
41	35B	G2	4	61.47	4.02	6.5	0.2
42	36	N2	4	16.37*	0.90	5.5	-40.6

** FOR CONFIDENCE INTERVAL 52.87 TO 60.68

TABLE NO. 5 SIGNIFICANCE LEVEL 0.05

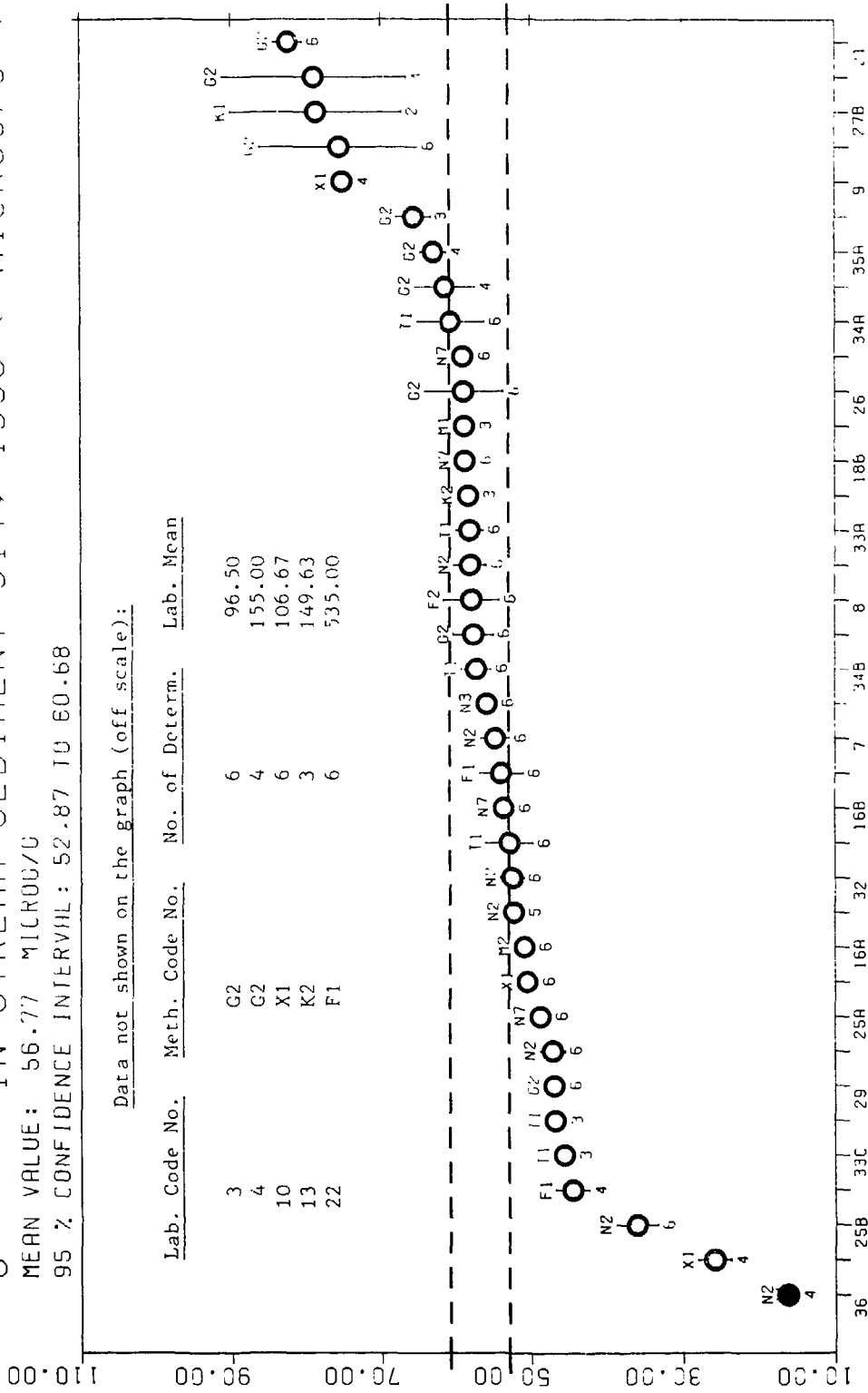
RESULTS OF INTERCOMPARISON FOR U-238 IN STREAM SEDIMENT-314, 1990

UNIT: MICROG/G

NO.	LAB. CODE NO.	METHOD CODE	NO.OF DETERM.	MEAN	STANDARD DEV.	
					ABS	REL %
1	37	G2	3	56.10	0.92	1.6

U IN STREAM SEDIMENT - 314, 1990 (MICROG/U)

MEAN VALUE: 56.77 MICROG/U
 95 % CONFIDENCE INTERVAL: 52.87 TO 60.68



LAB. CODE NO.

SUMMARY OF RESULTS OF THE INTERCOMPARISON STREAM SEDIMENT-314, 1990

RADIOACTIVE DETERMINED	RA-226	TH	U
UNIT	BQ/KG	MICROG/G	MICROG/G
NUMBER OF LABORATORY REPORTED RESULTS	27	32	42
INDIVIDUAL DETERMINATIONS	113	168	213
NUMBER OF LABORATORY ACCEPTED RESULTS	26	25	36
INDIVIDUAL DETERMINATIONS	111	131	184
TOTAL RANGE OF LABORATORY AVERAGES	455.07 - 1164.00	6.70 - 106.45	16.37 - 535.00
RANGE OF ACCEPTED LABORATORY AVERAGES	455.07 - 980.00	11.88 - 21.72	25.87 - 82.35
PERCENTAGE OF OUTLYING LABORATORIES	4	22	14
OVERALL MEAN OF ACCEPTED LABORATORY AVERAGES	732.49	17.77	56.77
STANDARD DEVIATION (S.D.)	136.04	2.46	11.52
REL%	18.6	13.9	20.3
STANDARD ERROR (S.E.)	26.68	0.49	1.92
REL%	3.6	2.8	3.4
CONFIDENCE INTERVALS FOR THE MEAN OF POPULATION FOR SIGNIFICANCE LEVEL .05	677.53 - 787.45	16.76 - 18.79	52.87 - 60.68

Table C

Recommended values and confidence intervals for Uranium, Thorium and Ra-226 in IAEA-314 (Stream Sediment).

Element	Recommended Value	Confidence Interval
Ra - 226	732 Bq/kg	678 - 787 (Class A)
Th	17.8 microg/g	16.8 - 18.8 (Class B)
U	56.8 microg/g	52.9 - 60.7 (Class B)

Confidence intervals are for significance level 0.05

Reference date: 30 January, 1988

List of participating laboratories for the IAEA's Intercomparison Study of the Stream Sediment (IAEA - 314) for Ra - 226, Thorium and Uranium

Algeria

Laboratoire des Techniques Nucleaires.CDTN.
"2 Bd. Frantz Fanon. Alger
B. Brahimi, I.Bacha

Australia

Government Chemical Laboratory. P.O. Box 594
Archerfield Q 4108
H.A. Olszowy.

Isotope Laboratory CRISO. Division of water resources.
Box 1666. Canberra
A.S. Murray, P.J. Wallibrink

The Alligator Rivers Region.
P.O. Box 387 Bondi Junction
N.S.W. 2022
R. Marten

Austria

IAEA Seibersdorf Laboratory, Chemistry Unit.
IAEA, Wagramerstr. 5, P.O.Box 100, A-1400 Vienna.
A. Ghods, E. Zepeda

Bundesversuchs und Forschungs-Anstalt Arsenal. Geotechnisches Institute,
Isotopengeophysik.
Faradaygasse 3
A - 1030 Wien
F.S. Maringer

Brasil

Instituto de Pesquisas Energeticas e Nucleares - IPEN/CNEN-S.P.
Caixa Postal 11049 - 05499. Sao Paulo
Vera L.R. Salvador

CNEN - Comissao Nacional de Energia Nuclear.
Laboratorio de Processos de Pocos de Caldas,
Caixa Postal 913. 37700 Pocos de Caldas
M. Nascimento

Comissao Nacional de Energia Nuclear
Instituto de Radioprotecao e Dosimetria.
Av. Americas Km 11.5 Barra da Tijuca
Rio de Janeiro Cx. P. 37750
M. Viammo

Instituto de Pesquisas Energe, Ticas E Nucleares
ME/MEC c.P. 11049 - 05499
Sao Paulo
S. Sarkis

Canada

Ecole Polytechnique. CP 6079 Sue "A"
Montreal Que H3C3A7
L. Zikovsky

Ministry of Labour. Radiation Protection Service.
81 Resources Road. Weston, ONT M9P 3TL
K. Gilmer

China

Chengdu College of Geology
Chengdu, Sichuan 610059
L. Guodong

Chengdu College of Geology
Chengdu, Sichuan 610059
T. Chunhan

Germany

Institute of Physics GKSS Research Centre
Box 1160. D-2054 Geesthacht
R. Pepelnik

Zentralstelle für Sicherheitstechnik. Abt. I.2
Ulenbergstr. 127-131. D-400 Dusseldorf
Fisher, Stockleth

Institute fuer Wasser-, Boden-, und Lufthygiene. Lab. B12
Corrensplatz 1. 1000 Berlin 33
H.U. Fusban

Central Institute of Nuclear Research
Rossendorf Box 19, Dresden 8051
W. Helbig

Central Institute of Nuclear Research
Rossendorf Box 19, Dresden 8051
B. Knobus, W. Rossbander

France

Cogema. SEPA/SAN B.P.
N71-87250 Bessines Sur Gartempe
M. Lemblin

Iraq

Activation Analysis Group. Nuclear Research Centre IAEC. Baghdad
P.O. Box 765
S.M. Al-Jobori A.M. Ali

Malaysia

Nuclear Energy Unit, Prime Minister's Department
Kompleks Puspati, Bangi
43000 Kajang
M.S. Hamzan

Republic of South Africa

Atomic Energy Corporation of SA. Building P-1600
Box 582. Pretoria 0001
C.B. Smit.

Romania

Institute of Physics and Nuclear Engineering. Section 6
Box 5206. Bucharest
A. Danis

Institute of Physics and Nuclear Engineering. Section 6
Box 5206. Bucharest
L. Dinescu

Institute of Physics and Nuclear Engineering. Section 6
Box 5206. Bucharest
M. Ciubotariu

Institute of Physics and Nuclear Engineering. Section 6
Box 5206. Bucharest
D. Razvan

Institute of Hygiene and Public Health
Radiation Hygiene Laboratory Str. Dr. Leonte 1-3
R - 76256, Bucharest 35
C. Milu, K. Gheorghe

Scotland

Department of Applied Chemical and Physical Sciences,
Napier Polytechnic of Edinburgh
Colinton Road, Edinburgh EH10 SDT
R.G. Pontin

Spain

Instituto "Pryma" - CIEMAT. Av. Complutense 22
28040 Madrid
J.P. Lopez.

United Kingdom

Atomic Weapons Establishment
Aldermaston, Reading, RG74PR
J. Johnson

United States of America

Laboratory of Inorganic and Nuclear Chemistry
NYSDOH, ESP, Wadsworth Center for Labs. and Research
Albany, New York 12201-0509
L. Husain

United State Department of Energy. Environmental Measurement Laboratory
376 Hudson Street. New York, NY 10014-3621
I.M. Fisenre

USSR

Polytechnik Institute Geological Faculty. Nuclear Geochim. Laboratory.
Lenin Pr. 30, 634004 Tomsk
E.G. Vertman

All Union Technology Institute of Geology, Geophysic and Information System
113 105 Moscow, Varshavskoe 8
A. Chernik

All Union Scientific Center of Radiation Medicine. AMS USSR
Meinikova 53. 252050 Kiev
I. Los

Yugoslavia

Laboratory for High Resolution Gamma Spectrometry
Department of Nuclear Physics. Institute "Jozef Stefan"
Jamova 39. POB 100. 61111 Ljubljana
M. Korun

Laboratorij za Nuklearnu Kemiju i Radiologiju. Insitute "Ruder Boskovic"
Bijenicka 54. Box 1016. 41000 Zagreb
S. Lulic

Zimbabwe

Institute of Mining Research. University of Zimbabwe
Box MP 167. MT Harrare
D. Mzemgeza

Method Code (without specification)

A1	Atomic absorption spectrometry. Flame
A5	Atomic absorption spectrometry. Hydride generation.
A6	Absorption technique
C4	Coulometry
E3	Emission spectrometry. DC ARC
E5	Inductively coupled Plasma. Simultaneous
G2	Gamma ray spectrometry without sample pre-treatment
G3	Gamma ray spectrometry with sample pre-treatment
G5	Gravimetric analysis
I1	Ion chromatography
N2	Neutron activation analysis. Thermal. Instrumental
N3	Neutron activation analysis. Epithermal. Instrumental
P4	Photometry
S2	Spectrophotometry
T1	Volumetric titration
T2	Potentiometric titration
T3	Colorimetric titration
T4	Turbidimetry
X1	X-Ray fluorescence analysis
Z	Unspecified method
K1	α -counting
R1	Radon emanation method