ANALYSIS AND ASSESSMENT OF THE RESULTS OF INTERCOMPARISON RUNS ON DETERMINATION OF SR-90, CS-137 AND PU-239/240 SPECIFIC ACTIVITY IN SOILS SAMPLED IN THE VICINITY OF "MAYAK"

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I INTRODUCTION

In correspondence with the decision of the Co-ordinating Board on territory rehabilitation of Ural region, intercomparison runs on determination of Sr-90, Cs-137 and Pu-239/240 specific activity in three type of soils, sampled in environmental surroundings of "Mayak", were carried out in 1993-1994 within the Radiation Measurements Quality Assurance Programme under supervision of Principal State Centre of Measurements Unity (VNII Mendeleev, St.-Petersburg), V.G.Khlopin Radium Institute (St.-Petersburg), "Mayak" (Chelyabinsk-45).

Soil samples have been treated and homogenised by special programme. Two of these samples were certified as Standard Reference Material of Russian Federation on specific activity of Sr-90, Cs-137, Pu-239/240. Certified values of radionuclides specific activity are shown in TABLE 1.

TABLE 1. Certified values of Cs-137, Sr-90 and Pu-239,240 specific activity in Standard Reference Materials of soil (Bq/kg).

SRM	Cs-137	Sr-90	Pu-239,240
SOIL-10	48.2 ± 2.2	16.7 ± 2.2	10.6 ± 2.4
SOIL-24	2440 ± 140	560 ± 70	2.2 ± 0.35
SOIL-3	157 ± 8	87 ± 9	1.7 ± 0.3

II. MAIN RESULTS OF THE INTERLABORATORY RUN.

26 laboratories including 6 foreign laboratories participated in the interlaboratory run

Programme of intercomparison runs implied determination of Sr-90, Cs-137, Pu-239/240 specific activity in two type of soils (SOIL-10, SOIL-24). In addition some laboratories analysed third type of soil - SOIL-3. As a rule, a number of parallel determinations for each radionuclide in soil sample was 3-6. On the basis of the reported data an mean weighted activity value (\overline{A}_i) for i-th laboratory was calculated together with appropriate value of standard deviation. $S_{\overline{A}}$. Those data, which did not

meet a condition $|A_i - \overline{A}| > 3 \cdot S_{\overline{A}}$, were rejected as outliers and not used for calculation of interlaboratory mean activity value. Main results of the interlaboratory run and some statistic for the measurement data obtained by the participants are given in TABLES 2-4.

TABLE 2. Summarised data for Sr-90 obtained by the participants of the interlaboratory run (Bq/kg).

SRM	Range of Lab Data A _i	$\begin{array}{c} \text{Lab Mean} \\ \text{Values and} \\ \text{SD } \overline{A}_i \pm S_{\overline{A}_i} \end{array}$	Number of data deviated from certified value less than 10%	Number of data deviated from certified value less than 25%
SOIL-10	13 - 57	18.6 ± 2.8	22%	67%
SOIL-3	60 -179	86.5 ± 9.6	38%	69%
SOIL-24	490 -950	550 ± 50	58%	84%

TABLE 3. Summarised data for Cs-137 obtained by the participants of the interlaboratory run (Bq/kg).

SRM	Range of Lab Data A _i	$\begin{array}{c} \text{Lab Mean} \\ \text{Values and} \\ \text{SD } \overline{A}_i \pm S_{\overline{A}_i} \end{array}$	Number of data deviated from certified value less than 10%	Number of data deviated from certified value less than 25%
SOIL-10	42 - 63	50.8 ± 1.4	75%	95%
SOIL-3	138 - 199	160.5 ± 6.5	80%	100%
SOIL-24	1840 - 2640	2480 ±140	85%	100%

TABLE 4. Summarised data for Pu-239,240 obtained by the participants of the interlaboratory run (Bq/kg).

SRM	Range of Lab Data A _i	$\begin{array}{c} \text{Lab Mean} \\ \text{Values and} \\ \text{SD \overline{A}_i \pm $S_{\overline{A_i}}$} \end{array}$	Number of data deviated from certified value less than 10%	Number of data deviated from certified value less than 25%
SOIL-10	2.6 - 11.9	8.4 ± 2.9	22%	44%
SOIL-3	0.3 - 3.3	1.67 ± 0.26	33%	53%
SOIL-24	1.4 - 3.0	2.4 ± 0.4	41%	71%

II. CONCLUSIONS

The following conclusions could be made from analysis of the reported data:

- in majority of the laboratories (17 from 20) semi-conductive gamma-spectrometric method for determination of Cs-137 allows to obtain reliable data both at low and high levels of Cs-137 content. For instance, mean weighted value of Cs-137 specific activity in sample SOIL-10 as received from results of interlaboratory runs is 49.8±3.2 Bq/kg. Certified value of the specific activity in the same soil is 48.8±2.2. So, within the measurement error (±4%) these results are satisfactorily agreed with each other.
- after evaluation of reported data on Sr-90, systematic errors were detected in 11 laboratories from 18. Analytical methods in four laboratories shall be tested thoroughly because these methods can not be applied for environmental control on Sr-90 content in soils in range 15-100 Bq/kg and lower.
- as a whole, analysis on Pu content resulted in rather poor quality especially in case of SOIL-10 and SOIL-3. Eight laboratories from 18 reported non confident data for SOIL-10 and 8 laboratories from 15 reported non confident data for SOIL-3. One of the main reason for that (in case of SOIL-10) is application of analytical methods based on partial dissolution of (the many times leaching by acid mixture) of soil subsample. Our special investigations have shown that Pu exists in poorly soluble form in SOIL-10. Total dissolution (\geq 99.5%) of the subsample is required for analysis of this type of soil because only in doing this confident data can be obtained. SOIL-3 contains rather low concentrations of Pu (1.7±0.3 Bq/kg) and sensitivity of analytical methods in some laboratories are not enough.
- in total, the number of confident determinations of radionuclides specific activity in three type of soil from Ural region is as it follows:

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Cs-137 - 85 % (47 from 55)
Sr-90 - 75 % (40 from 53)
Pu-239,240 - 60 % (30 from 50)
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- the laboratories who have shown positive results in the intercomparison runs could certify their analytical methods in D.I.Mendeleev VNIIM together with V.G.Khlopin Radium Institute.

The laboratories who have reported non confident data may correct their analytical methods accounting for results of intercomparison runs and certified values of soil SRMs.