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# Certificate of Measurement

## CERTIFIED REFERENCE MATERIALS

### IARMA-002, IARMA-003 and IARMA-004

#### GAMMA-EMITTING RADIONUCLIDES IN WATER

##### Certified Values of Massic Activity

Radionuclide	IARMA-002		IARMA-003		IARMA-004	
	Certified value <sup>1</sup>	Expanded uncertainty <sup>2</sup>	Certified value <sup>1</sup>	Expanded uncertainty <sup>2</sup>	Certified value <sup>1</sup>	Expanded uncertainty <sup>2</sup>
	[Bq kg <sup>-1</sup> ]					
Co-57	3.08	0.10	6.83	0.22	16.54	0.52
Co-60	3.23	0.10	7.99	0.24	19.54	0.60
Ba-133	2.52	0.06	4.86	0.15	10.43	0.31
Cs-134	2.99	0.10	6.31	0.20	13.85	0.40
Cs-137	2.30	0.10	13.98	0.28	13.10	0.39
Eu-152	4.07	0.10	6.05	0.20	14.19	0.43
Pb-210	16.20	0.50	28.60	0.80	53.30	1.60
Am-241	2.70	0.10	7.10	0.20	16.24	0.49

<sup>1</sup>The certified value is calculated from the certified activity values assigned to each standard source, taking into account the successive gravimetric dilution steps, the mass of the spike mixture and the amount of water being spiked as determined from weighing. The certified values are traceable to the SI units.

<sup>2</sup> Combined expanded uncertainty [1] with a coverage factor  $k = 2$  consists of three major components of uncertainties associated with the certified standard sources, weighing for dilution and stability of water samples.

\*Reference date for decay correction: 01-November-2013

Issue date of Certificate: 31-March-2014

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Chief Executive Officer

Origin and preparation of the material

De-mineralized water, sourced in one batch, was used to prepare the water materials. The source water was acidified and then analysed for the radionuclides of interest.

A portion of the blank water was then spiked with an appropriate amount of a mixture of single radionuclide certified standard solutions, traceable to the international standards of radioactivity through the Czech Metrological Institute. Table 1 shows the certified standard solutions of each radionuclide used in spiking the water.

Table 1. Certified standard solutions of each radionuclide

Nuclide	Certificate number	$t_{1/2}$ [days]	Massic activity [Bq g <sup>-1</sup> ]	Standard uncertainty [Bq g <sup>-1</sup> ]
Co-57	9031-OL- 570/13	271.26	59710	180
Co-60	9031-OL- 569/13	1925.4	73761	590
Ba-133	9031-OL- 571/13	3897	101300	405
Cs-134	9031-OL- 566/13	753	46652	140
Cs-137	9031-OL- 567/13	11019	103802	520
Eu-152	9031-OL- 572/13	4858	88208	440
Pb-210	9031-OL- 565/13	8108	32897	330
Am-241	9031-OL- 568/13	157800	112524	225

Reference date: 10-October-2013

#### **Intended use**

Based on well-defined metrological characteristics (metrological traceability of the certified values and associated uncertainties) IARMA-002, 003 & 004 are suitable for quality assurance and quality control purposes and for method development and all aspects of analytical method validation, including potential bias and efficiency calibration evaluation, and for training purposes. IARMA-002, 003 & 004 could also be used as test items for inter-laboratory comparisons and/or proficiency tests.

#### **Instructions for use**

IARMA-002, 003 & 004 Certified Reference Materials are supplied in 250 g units and the material homogeneity is guaranteed if a minimum test portion of 25 g is used.

When the IARMA CRMs are used for evaluation of method bias, the method can be considered free of significant bias at a confidence level of 99% if the following criterion is fulfilled:

$$A1 \leq A2 \quad \text{where: } A1 = |\text{Value}_{\text{reference}} - \text{Value}_{\text{measured}}|; \quad A2 = 2.58 \times \sqrt{u_{\text{reference}}^2 + u_{\text{measured}}^2}$$

Value<sub>reference</sub>, u<sub>reference</sub>: Reference value and its associated standard uncertainty of the studied analyte from the Certificate.

Value<sub>measured</sub>, u<sub>measured</sub>: Mean value of at least 7 independent measurement results and its associated standard uncertainty of the studied analyte.

#### **Homogeneity test of the material**

The spiked water was homogenised using a manual mixer in a 200-liter tank and then bottled into 250 ml portions in HDPE bottles. The total mass of the labelled bottle was recorded for further control of losses. Three bottles were selected randomly from each dilution step. Three test portions of 100 g were taken from each of the selected bottles and analysed using gamma-ray spectrometry. It was found that the relative standard deviations of measurement results (n=3) of all analytes were below the method repeatability relative standard deviation, demonstrating satisfactory homogeneity of the material.

#### **Verification of the certified values**

The final certified value for each radionuclide in the water material, IARMA-002, 003 & 004, was calculated from the certified value assigned to each certified standard solution radionuclide, taking into account the successive gravimetric dilution steps, the mass of the spike and the amount of water spiked.

The combined standard uncertainty included three major components of uncertainties associated with the certified standard solution, weighing for dilution and stability of water material.

To confirm the certified values of the IARMA-002, 003 & 004 CRMs, three bottles from each material were analysed at the Josef Stefan Institute Laboratory in Ljubljana, Slovenia.

The degree of equivalence between the certified values calculated from the dilution of the certified standard solution and their actual measured values was calculated according to the BIPM approach [4] which takes into consideration the uncertainty of measured values using formulas 1 and 2. Table 2 lists the degree of equivalence of the activity concentrations of the radionuclides in the IARMA water CRMs.

For this purpose, the absolute bias  $B_i$  is calculated according to formula (1) then the propagated expanded uncertainty of both measured and calculated activity concentrations  $U(B_i)$  is calculated as per formula (2).

$$B_i = ABS(x_i - x_{CRM}) \quad (1)$$

$$U(B_i) = 2 * \sqrt{u(x_i)^2 + u(x_{CRM})^2} \quad (2)$$

Where:

$x_i$  is the measured activity concentration (i = 1 to n),

$x_{CRM}$  is the reference value calculated from the dilution of the standard source,

$B_i$  is the difference between the measured and calculated activity concentrations,

$u(x_i)$  is the standard uncertainty of the measured activity,

$u(x_{CRM})$  is the standard uncertainty associated with the certified value ,

$U(B_i)$  is the expanded uncertainty (k = 2) of the difference  $B_i$  at a 95% level of confidence.

The reference value calculated from the dilution is confirmed and considered equivalent to the measured value at a confidence level of 95% if the Degree of Equivalence (DoE) is smaller than 2:

$$DoE = \frac{B_i}{U(B_i)} \quad (3)$$

Table 2. Degree of equivalence between the certified values as calculated and their measurement results

Radio-nuclide	Water IARMA-002			Water IARMA-003			Water IARMA-004		
	$d_i$	$U(d_i)$	DoE	$d_i$	$U(d_i)$	DoE	$d_i$	$U(d_i)$	DoE
	[Bq/kg]			[Bq/kg]			[Bq/kg]		
Co-57	0.09	0.23	0.39	0.46	0.46	1.01	0.90	1.09	0.82
Co-60	0.03	0.24	0.12	0.08	0.47	0.17	0.20	1.18	0.17
Ba-133	0.01	0.17	0.06	0.15	0.26	0.57	0.30	0.54	0.55
Cs-134	0.1	0.23	0.43	0.01	0.38	0.03	0.40	0.84	0.47
Cs-137	0.03	0.13	0.23	0.4	0.67	0.59	0.10	0.73	0.14
Eu-152	0.05	0.35	0.14	0.08	0.50	0.16	0.30	0.82	0.37
Pb-210	1.1	2.02	0.55	2.7	3.63	0.74	3.00	6.53	0.46
Am-241	0.04	0.15	0.27	0.08	0.35	0.23	0.10	0.89	0.11

### **Statement on metrological traceability and uncertainty of assigned values**

The certified value assigned to the measurands of IARMA-002, 003 & 004 is massic activity, expressed in the derived SI unit Bq kg<sup>-1</sup>. The certified values were calculated from the certified values assigned to each certified standard solution radionuclide used in the spike; metrological traceability to the SI Units was provided for all used certified standard solution.

### **Commutability of the Certified Reference Materials**

The user of the IARMA-002, 003 & 004 CRMs should be aware of the aspect of commutability when samples of different density or chemical composition are analysed.

### **Handling and storage**

It is recommended that the original unopened bottle be stored securely in a refrigerator at 5±2 °C. It is recommended to avoid direct exposure to sunlight or to a source of heat.

### **Issue and expiry date**

Based on experience with similar materials and the stable and controlled storage conditions and performed stability study, the certified values for the studied measurands are valid until 20-June-2026, provided the original bottle is handled and stored in accordance with the provided instructions.

### **Legal disclaimer**

IARMA Limited makes no warranties, expressed or implied, with respect to the data contained in this Certificate and shall not be liable for any damage that may result from the use of such data.

### **Compliance with ISO Guide 31:2015**

The content of this Certificate is in compliance with the ISO Guide 31:2000: Reference materials - Content of certificates and labels [4].

### **Note**

Certified values as stated in this Certificate may be updated if more information becomes available. Users of these CRMs should ensure that the Certificate in their possession is current. The current version can be found on the IARMA Limited website (<http://www.iarma.co.uk>).

For further information regarding this material, please contact [office@iarma.co.uk](mailto:office@iarma.co.uk)

### **REFERENCES**

[1] Joint Committee for Guides in Metrology (JCGM), JCGM 100:2008 (GUM with minor corrections), Evaluation of measurement data - Guide to the expression of uncertainty in measurement. ([http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf))

[2] International Organization for Standardization, ISO Guide 35:2006(E), Reference materials – General and statistical principals for certification.

[3] International Organization for Standardization, ISO Guide 13528:2005(E), Statistics methods for use in proficiency testing by interlaboratory comparisons.

[4] International Organization for Standardization, ISO Guide 31: 2015(E), Reference materials - Content of certificates and labels.

[5] Shakhashiro, A., Toerveyi, A., Gaudino, S., Rosamilia, S., Belli, M., Turk, G. C., CCQM Key Comparison CCQM-K75: Determination of Toxic Metals in Algae, Metrologia, v. 48 (2011), 49 p., doi: 10.1088/0026-1394/48/1A/08002.