



**IARMA Limited**  
Naver House, Naver Road  
Thurso, Caithness, KW14 7QA  
United Kingdom  
Email: office@iarma.co.uk  
www.iarma.co.uk

The International Atomic Reference Material Agency

## Certificate of Analysis

### CERTIFIED REFERENCE MATERIAL

IARMA-006, IARMA-007, IARMA-008 and IARMA-009

#### TRITIUM in WATER

Certified Value: Derived Massic Activity

CRM Code	Activity	Uncertainty*	Activity	Uncertainty
		[TU]		[Bq/kg]
IARMA-006	6.1	0.2	0.72	0.02
IARMA-007	21.8	0.5	2.6	0.08
IARMA-008	109.1	3.0	13.0	0.4
IARMA-009	213.1	5.2	25.4	0.8

Uncertainty: Combined standard uncertainty expressed at  $1\sigma$  level and consists of the uncertainties associated with the certified standard source used for spike, weighing for dilution and stability of the water.

**Reference date for decay correction: 15-May-2014**

The tritium massic activity is related to the tritium ratio via the following relationship: 1 Bq/kg corresponds to  $8.390 \pm 0.015$  TU [1, 2], using the new value for the tritium half-life [3] and the CODATA 1998 value for the Avogadro constant [4].

J. P. Doherty  
Chief Executive Officer

## Preparation of the CRMs

The test items were prepared by accurate gravimetric dilution of the NIST tritiated water standard SRM4926E with accurately weighed portions of the dead water IARMA-005 [5].

The spiked water was homogenised using a manual mixer in a 75 litre 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 test portions of 250 g were analysed by LSC with enrichment and direct measurement.

The Z-test demonstrated an acceptable level of homogeneity as all calculated Z-test according to formula (1) were below 2.

$$Z_{TEST} = (x_1 - x_2) / \sqrt{u(x_1)^2 + u(x_2)^2} \quad (1)$$

Where:

$x_1 - x_2$  is the difference between two independent measurement results of two different bottles,

$\sqrt{u(x_1)^2 + u(x_2)^2}$  is the propagated uncertainty of both measurement results.

In addition, it was found that the relative standard deviation of the homogeneity test measurement results was comparable to the method repeatability relative standard deviation, demonstrating that the prepared test items are homogenous.

The stability of the test items was tested by analysing the test items immediately after the preparation and after at intervals of three months of storage at room temperature for one year. The results demonstrated that the activity concentration of H-3 is stable.

Moreover, the total mass of each bottle was registered on the bottle label for evaporation control and there was not any significant loss of water mass observed due to evaporation during a one week storage at  $45 \pm 2$  °C.

## Verification of the reference values

The final reference values of tritium massic concentration in the test items, were calculated from the certified value listed in the certificate of the NIST standard solution of tritium SRM4926E, taking into account the successive gravimetric dilution steps, the mass of the spike and the amount of water spiked. The combined standard uncertainty includes three major components of uncertainties associated with: the uncertainty of the certified value of the standard solution, weighing for dilution and stability of water material. A minor source of uncertainty was considered which arises from the uncertainty associated with the correction of the reference values to the PT reference date (15 May 2014) using the half-life  $4500 \pm 8$  days.

To confirm the assigned reference values of the test items, three bottles from each were analysed. The Z-test between the assigned reference activity calculated from the dilution of the certified standard solution and their actual measured values was calculated according to the formula (1) which takes into consideration the uncertainty of measured values. Table 1 lists the Z-test values which demonstrate a good agreement between the measured values and the calculated ones.

TABLE 1. Comparison between the calculated reference activity and the measurement activity of tritium in water test items based on z-test

	<i>Measured Activity</i>	<i>Unc.</i>	<i>Calculated Reference Activity</i>	<i>Unc.</i>	<i>Z-Test</i>
	[TU]		[TU]		
IARMA-006	7.0	0.7	6.1	0.2	1.24
IARMA-007	22.1	0.9	21.9	0.5	0.19
IARMA-008	110.9	3.3	110.2	3.0	0.16
IARMA-009	213.7	6.4	214.8	5.2	-0.13

### **Intended use**

These CRMs are suitable for quality assurance and quality control purposes and for method development to monitor the method performance and to check the calibration of the system and for all aspects of analytical method validation, including verification of method trueness and precision, and for training purposes.

### **Handling and storage**

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

### **Issue and expiry date**

The issue date of this Certificate is 20-February-2015. Based on experience with similar materials and the performed stability study, the certified value for tritium is valid until 20-June -2029, 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 31:2000**

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

### **Note**

The certified value 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] GRÖNING, M., ROZANSKI, K., Uncertainty assessment of environmental tritium measurements in water. Accred. Qual. Assur. 8(8) (2003) 359-366.

[2] Rozanski, K., Gröning, M. Tritium assay in water samples using electrolytic enrichment and liquid scintillation spectrometry. In: Proceedings of a Consultants Meeting on Quantifying Uncertainty in Nuclear Analytical Measurements, 11-14 May 1998 in Vienna: International Atomic Energy Agency. (2004) p. 195-217.

[3] Lucas, L.L., Unterweger, M.P., Comprehensive review and critical evaluation of the half-life of tritium. J. Res. Nat. Inst. Stand. Technol. 105 (2000) 541-549.

[4] Mohr, P., Taylor, B.N., CODATA recommended values of the fundamental physical constants: 1998. Review of Modern Physics 72(2) (1998) 351-495.

[5] Certificate of analysis IARMA-006, <http://www.iarma.co.uk>.

**END of the certificate**