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Certified Reference Material IAEA-418: I-129 in Mediterranean Sea Water

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FOREWORD

Our society is attaching increasing importance to the study and assessment of the state and health of the environment. Organizations involved in such activities rely on the quality of the information provided and, ultimately, on the precision and accuracy of the data on which the information is based. Many laboratories are involved in the production of environmental data in many cases leading to wider assessments. These laboratories may develop and validate new analytical methods, study the environmental impact of human activities, provide services to other organizations, etc. In particular, laboratories are providing data on levels of radioactivity in a variety of marine matrixes such as water, suspended matter, sediments and biota. Because of the need to base scientific conclusions on valid and internationally comparable data, the need to provide policy makers with correct information and the need for society to be informed of the state of the environment, it is indispensable to ensure the quality of the data produced by each laboratory.

Principles of good laboratory practice require both internal and external procedures to verify the quality of the data produced. Internal quality is verified in a number of ways such as the use of laboratory information systems, keeping full records of equipment performance and standardization of analytical procedures. External quality can also be ascertained in a number of ways, notably accreditation by an external body under a defined quality scheme but also, amongst others, the use of internationally accepted calibration standards that are traceable to the SI international system of units, the participation in interlaboratory comparisons or the regular use of reference materials to test laboratory performance.

The Radiometrics Laboratory of the IAEA Marine Environment Laboratories has been providing quality products for the last 40 years which include the organization of interlaboratory comparisons, proficiency tests, production of reference materials and certified reference materials, and training [1]. More than 40 reference materials have been produced, which include a wide range of marine sample matrices and radionuclide concentrations.

As part of these activities, a new interlaboratory comparison was organized to provide the participating laboratories with the possibility to test the performance of their analytical methods for ^{129}I on a Mediterranean sea water sample. The material was designed for the analysis of low level ^{129}I in sea water. The sample, after successful certification, has been issued as a certified reference material for ^{129}I in sea water.

Laboratories were informed that, after the completion of the exercise, an IAEA report describing the results of the interlaboratory comparison would be issued, including their identities, but that the results would remain anonymous.

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CONTENTS

1. INTRODUCTION

The accurate and precise determination of radionuclide concentrations in marine samples are important aspects of marine radioactivity assessments and the use of radionuclides in studies of oceanographic processes. To address the problem of data quality, the IAEA Marine Environment Laboratories (IAEA-MEL) in Monaco regularly conduct interlaboratory comparisons on radionuclides in marine samples as part of its contribution to the reference materials activities [1, 2].

Following the discussion during the AMS (accelerator mass spectrometry) meeting held in Vienna in 2000, the AMS scientific community strongly recommended the production of a low level ^{129}I sea water reference material.

This report describes the results obtained from 9 laboratories on the determination of ^{129}I in a Mediterranean sea water sample.

2. SCOPE OF THE INTERLABORATORY COMPARISON

This reference material was characterised through an interlaboratory comparison exercise for the measurement of ^{129}I in sea water at environmental levels. Participating laboratories were requested to determine the ^{129}I concentration using adequate techniques such as AMS.

The sample, after successful certification following ISO Guide 35 [3], has been issued as a certified reference material for ^{129}I in sea water.

3. DESCRIPTION OF THE MATERIAL

About 600 L of surface water were collected at the Dyfamed station (43° 25.117' N – 07° 50.040' E) in the Mediterranean sea on 18 February 2001. The sample was filtered through a membrane filter with a 0.45 μm pore size. No preservatives were added to the sample.

4. HOMOGENEITY

The sample was initially transferred into three 500 L containers and then homogenized by using pumps to mix water between the containers back and forth for more than 8 hours. The samples were bottled in 1 L air-tight polyethylene bottles and labelled with the code IAEA-418 for a total of 200 bottles. These bottles were then stored at ambient temperature in dark conditions.

5. SAMPLE DISPATCH AND DATA RETURN

Each participant received 2 L of the seawater sample. The following information was requested:

- number of analyses;
- net activity concentration (i.e. corrected for blank, background etc.) and expressed in atoms L^{-1} or Bq L^{-1};
- estimation of the combined uncertainty;
- description of chemical procedures and counting equipment;
- standards and calibrators used;
- chemical recoveries, counting time, half-life.

The samples were distributed to 12 laboratories in October 2001. Taking into account the difficulty of the technique used and the limited number of laboratories equipped with AMS, the exercise was extended to other laboratories. A total of 9 laboratories sent their final reports. The list of contributing laboratories can be found at the end of this report.

6. EVALUATION OF RESULTS

6.1. Data treatment

The results are shown under their laboratory code numbers in Table 1. Laboratory means were calculated when necessary from individual results and are given either as arithmetic means with corresponding standard deviation when more than two results were reported, or as weighted means with weighted uncertainties in the case of only two results reported. All values were rounded off to the most significant number.

6.2. Statistical evaluation

The principles and applications of the statistical procedure used for the evaluation of data have been described in a previous report [4]. Calculations are based on the assumption of non-parametric distribution of data to which distribution-free statistics are applicable. The 'less than' values are segregated from the results and the remaining values are checked for the presence of outliers using a box and whisker plot test. Outliers, if any, are identified in the tables with an asterisk. Median values are calculated from all results passing the test and are considered to be the most reliable estimates of the true values. Confidence intervals were taken from a non-parametric sample population. They represent a two-sided interval representing 95% confidence limits. Expanded uncertainty with a coverage factor of k=2, corresponding to a level of confidence of about 95%, was also calculated according to the ISO-1993 and NIST Guidelines [5, 6].

Following the International Union of Pure and Applied Chemistry (IUPAC) [7] and the International Organization for Standardization (ISO) [8] recommendations for assessment of laboratory performance, the Z-score methodology was used for the evaluation of the interlaboratory comparison results. The performance of a laboratory was considered to be acceptable if the difference between the robust mean of the laboratory and the assigned value is less than or equal to two. The analysis is regarded as being out of control when $|Z|>3$.

Following the International Committee of Weights and Measures (CIPM) recommendation [9], the degree of equivalence of reported results was also included in the data evaluation process. It permits to verify if the measurement reported is consistent with the key comparison reference value.

6.3. Explanation of tables

6.3.1. Laboratory code

Each laboratory was assigned an individual code number to ensure anonymity. Laboratories were informed that, after the completion of the exercise, an IAEA report describing the results of the interlaboratory comparison would be issued, including their identities, but that the results would remain anonymous.

6.3.2. Method used

Most participants used AMS (accelerator mass spectrometry) with prior radiochemical enrichment of iodine. One laboratory used NAA (nuclear activation analysis) but reported results as LLD (lower limit of detection).

The chemical method most frequently used by participants to determine ^{129}I concentration in sea water was the following [10, 11]: 200 to 500 ml of water was filtered through a 0.45 μm membrane filter and ^{127}I was added as a carrier. The solution was then reduced with $NaHSO_3$ to convert iodate to iodide and acidified to pH 2. Iodine was extracted with $CHCl_3$ or CCl_4, back extracted into water, precipitated as AgI, washed twice with distilled water and dried at 60°C. The AgI was mixed with niobium powder and pressed into cupper holders for the AMS measurement.

The procedure for the AMS measurement of $^{129}I/^{127}I$ can be summarized as follows [12]: using a Cs^+ sputter source, a beam of negative iodine ions is extracted from the AgI and Nb mixture target and is energy analyzed. ^{129}I and ^{127}I are separated in the first bending magnet and injected sequentially into a tandem accelerator. At the terminal of the tandem, negative iodine ions are changed to positive ions by an electron stripper, which are then further accelerated. Following acceleration, selected positive iodine ions are analyzed by mass spectrometry. The ^{127}I current beam is measured in a Faraday cup and ^{129}I ions are counted in a gas ionization detector. The use of negative ions in the ion source eliminates the interferences from the isobar ^{129}Xe because it does not form stable negative ions. The use of a tandem accelerator eliminates the interferences from molecules of mass 129 because they are fragmented in the electron stripper process of charge changing at the terminal of the tandem. These two features make the AMS technique highly sensitive for the measurement of long lived radioactive isotopes.

Background and memory effects are evaluated through carrier iodine blanks that produce a procedural background $^{129}I/^{127}I$ value less than 10^{-13}. The machine background is evaluated through the use of a natural AgI (iodargyrite) which results in a value of 10^{-14}. The $^{129}I/^{127}I$ of the samples were about 150 times higher than the natural level, thus resulting in a negligible (<1%) background correction. Finally, the ^{129}I concentration in the original sample is calculated from the volume of sample used, the total iodine concentration in the original sample, the amount of ^{127}I carrier and the $^{129}I/^{127}I$ ratio determined in the AMS measurement.

6.3.3. Number of results

The number of determinations corresponds to the number of individual results from which the laboratory mean was calculated. When no mention was made in a participant's report as to the number of measurements made, it was assumed to be one.

6.3.4. Concentration

The concentration corresponds to the arithmetical or weighted mean computed from all the individual results obtained from the participants with the corresponding standard deviation or weighted uncertainty expressed in atoms L^{-1} (Table 1) or in Bq L^{-1} (Table 2).

A summary of the recommended value expressed in atoms L^{-1} and Bq L^{-1} with confidence intervals and their expanded uncertainties is given in Table 3.

6.3.5. Degree of equivalence of the reported results

To compare the relationship between the reported results and their uncertainties, an estimator ε (degree of equivalence of a reported results) was calculated by rating the difference between the reported value and the reference value (median) to the total propagated uncertainty of this difference with a coverage factor of two (95% confidence level).

$$\varepsilon = \left| Value_{reference} - Value_{reported} \right| / \; 2 \times \sqrt{unc^2_{reference} + unc^2_{reported}}$$

A smaller ε indicates a better relationship. The ε values, listed in Table 4, show that 100% of the reported results have an ε value less than 2. It can be concluded that there is a high level of commutability of the reported results.

TABLE 1. RESULTS FOR ^{129}I IN IAEA-418 (in atoms L^{-1})

Lab. code	Method code	No. of results	Volume (ml)	^{129}I ($\times 10^8$ atoms L^{-1})
1	AMS	3	500	2.3 ± 0.1
2	AMS	4	500	2.7 ± 0.3
3	AMS	1	350	2.7 ± 0.7
4	AMS	4	200	2.20 ± 0.05
5	AMS	2	200	2.8 ± 0.1
6	AMS	2	200	2.3 ± 0.2
7	AMS	6	400–500	2.16 ± 0.05
8	NAA	1	-	LLD
9	AMS	1	200	11.9 ± 1.4*
Number of reported lab. means				9
Number of accepted lab. means				7
Median				2.3
Confidence interval ($\alpha = 0.05$)				2.2–2.8
Expanded uncertainty (k=2)				0.2

* Outlier value

TABLE 2. RESULTS FOR ^{129}I IN IAEA-418 (in Bq L^{-1})

Lab. code	Method code	No. of results	Volume (ml)	^{129}I ($\times 10^{-7}$ Bq L^{-1})
1	AMS	3	500	3.2 ± 0.2
2	AMS	4	500	3.7 ± 0.4
3	AMS	1	350	3.8 ± 1.0
4	AMS	4	200	3.1 ± 0.1
5	AMS	2	200	3.9 ± 0.1
6	AMS	2	200	3.2 ± 0.3
7	AMS	6	400–500	3.02 ± 0.07
8	NAA	1	1	LLD
9	AMS	1	200	15.5 ± 1.9*

Number of reported lab. means	9
Number of accepted lab. means	7
Median	3.2
Confidence interval ($\alpha = 0.05$)	3.0–3.9
Expanded uncertainty (k=2)	0.3

* Outlier value

TABLE 3. RECOMMENDED VALUES FOR IAEA-418 (REFERENCE DATE: 18 FEBRUARY 2001)

Measurand	Certified value	Expanded uncertainty (k=2)
^{129}I concentration (atoms L^{-1})	2.3×10^{8}	0.2×10^{8}
^{129}I activity concentration[£] (Bq L^{-1})	3.2×10^{-7}	0.3×10^{-7}

[£] Half-life of ^{129}I is $(1.57 \pm 0.04) \times 10^{7}$ yr [14]

TABLE 4. DEGREE OF EQUIVALENCE ε FOR THE REFERENCE MATERIAL IAEA-418 ^{129}I IN MEDITERRANEAN SEA WATER

Lab. code	ε for ^{129}I concentration (atoms L^{-1})	ε for ^{129}I activity concentration (Bq L^{-1})
1	0.00	0.00
2	0.58	0.57
3	0.30	0.29
4	0.19	0.18
5	1.25	1.17
6	0.03	0.03
7	0.29	0.27

6.4. Explanation of figures

Figure 1 shows the results of the outlier test using a box and whisker plot. Seven values passed this test and were accepted for calculation of the medians, confidence intervals and expanded uncertainties.

Figure 2 presents the data evaluation with the corresponding standard deviation or weighted uncertainty. Also shown are:

(i) the median (full line) and corresponding confidence interval (dashed horizontal lines);
(ii) the limit for accepted laboratory means (vertical line).

The performance of laboratories in terms of accuracy was expressed by Z-scores in Figure 3. The distribution of Z-scores is symmetric with their values less than 2, indicating that the overall performance of the laboratories was satisfactory.

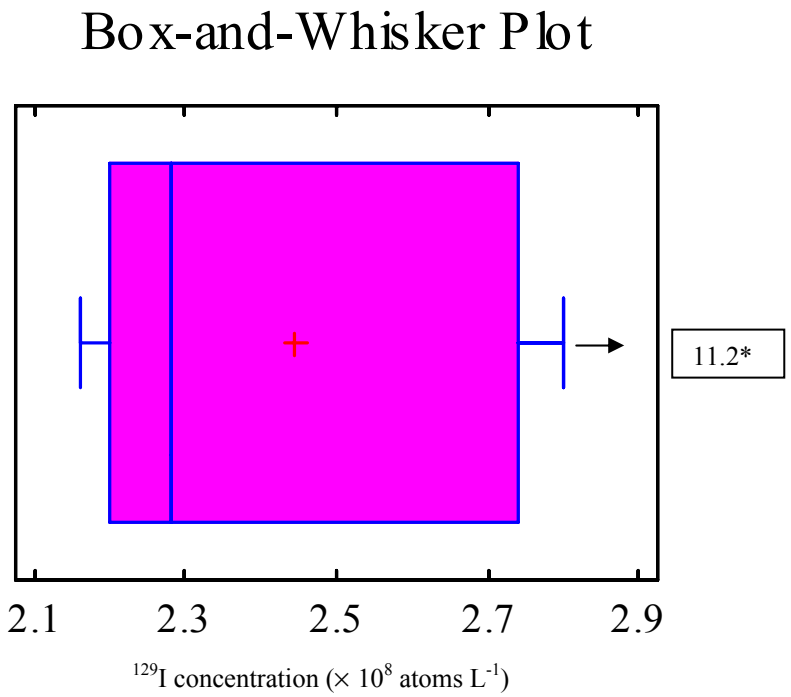

FIG. 1. Outlier test results for ^{129}I in IAEA-418.

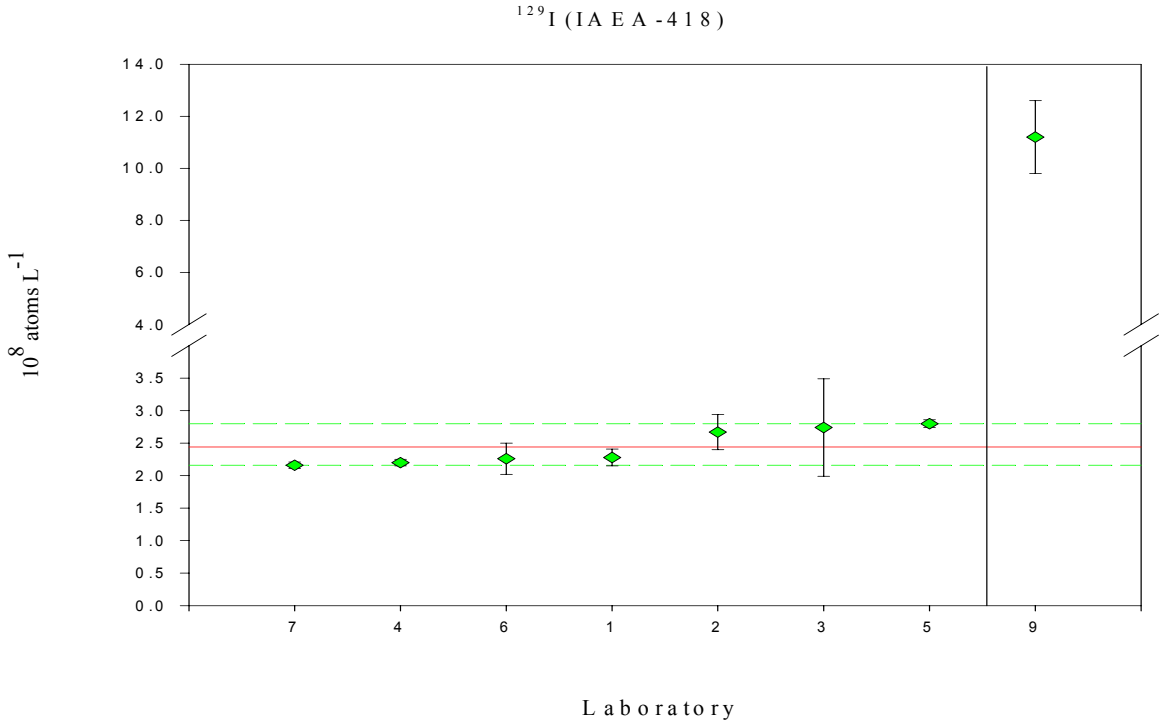

FIG. 2. Data evaluation for ^{129}I in IAEA-418.

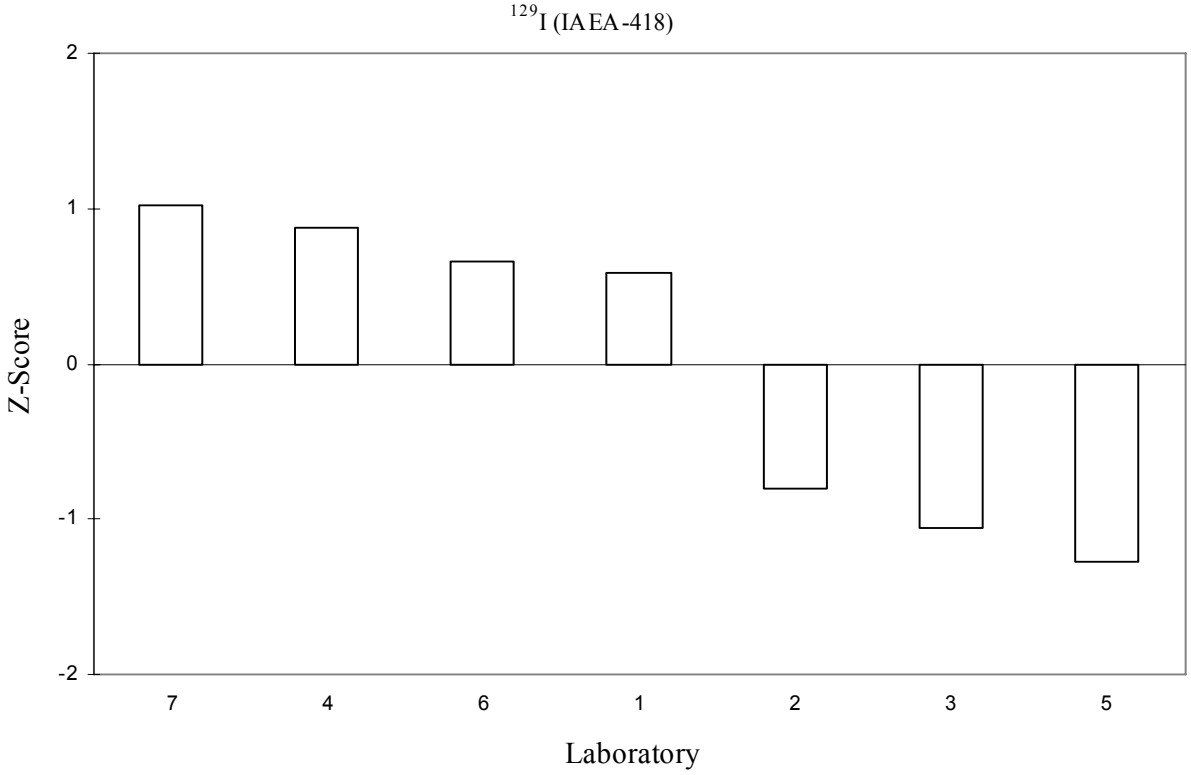

FIG.3. Z-score for ^{129}I in IAEA-418.

6.5. Criteria for certification and metrological traceability

The median values of the data were considered as the certified values as [1]:

- At least 5 laboratory means were available.
- The relative uncertainty of the median did not exceed 30% for an activity lower than 10μBq L^{-1}.

The median concentrations for the sets of individual data — after rejection of outliers — were chosen as the best estimations of the property values [1–4, 13] and are reported as certified values. Expanded uncertainties with a coverage factor of k=2, corresponding to a level of confidence of about 95%, were calculated according to the ISO-1993 and NIST Guidelines [5, 6]. Evidence on metrological traceability to the SI Units was provided by all laboratories and is summarized in Table 5.

TABLE 5. METROLOGICAL TRACEABILITY OF REPORTED RESULTS

Lab. code	Instrument	Standards and calibrators used
1	AMS-HVEE 3MV accelerator	NIST 4949C
2	AMS-HVEE 1MV accelerator	NIST 3230
3	AMS-NEC 3MV pelletron accelerator	AgI standards (Purdue University, USA)
4	AMS-5MV pelletron tandem accelerator	NIST 4949C
5	IsoTrace heavy element AMS	AgI material ISO-2
6	IsoTrace heavy element AMS and AMS-HVEE 1MV accelerator	AgI material ISO-2 and NIST 3230
7	AMS-0.5MV tandem accelerator	NIST 4949B

7. FINDINGS AND CONCLUSIONS

In this interlaboratory comparison, 9 laboratories reported concentrations of ^{129}I in a sea water sample from the Mediterranean sea (IAEA-418). The median concentrations for the sets of individual data — after rejection of outliers — were chosen as the best estimations of the property values and are reported as certified values. These certified values and their expanded uncertainties can be found in Table 3.

This certified reference material IAEA-418 is intended to be used for quality assurance purposes in the determination of ^{129}I in sea water samples, including development and validation of analytical procedures, preparation and testing of reference methods, quality control, and training of analysts.

This material is not to be used as calibrant.

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