

## INTERCOMPARISON EXERCISE FOR RADIONUCLIDES IN MEDITERRANEAN MUSSEL (IAEA-437)

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### Abstract

An intercomparison exercise for natural and anthropogenic radionuclides was organized using a material prepared from the soft tissues of mussels collected in the vicinity of the Grand Rhone river mouth. Seven out of the 15 laboratories participating in the CIESM Mediterranean Mussel Watch Program reported results. Based on these results and on those received from 9 additional laboratories worldwide, recommended values were derived for <sup>40</sup>K, <sup>234</sup>U and <sup>239+240</sup>Pu and information values for <sup>137</sup>Cs, <sup>210</sup>Pb(Po), <sup>226</sup>Ra, <sup>228</sup>Th, <sup>232</sup>Th, <sup>235</sup>U, <sup>238</sup>U and <sup>241</sup>Am. Further work is underway for the certification of this intercomparison material as IAEA Reference Material.

**Keywords :** Radionuclides, Monitoring, Pollution, Instruments And Techniques.

The International Atomic Energy Agency's Marine Environment Laboratory (IAEA-MEL) conducted 44 intercomparison exercises and produced 39 marine Reference Materials (RMs) over the past three decades [1]. This programme responds to an increasing interest of data producers in the quality of analytical data, which parallels the increased public interest in environmental issues and the statutory quality demands placed by governmental institutions on food and environmental data producers. The trends in marine environmental contaminant levels, the emergence of new contaminants and, consequently, of new or improved analytical techniques, set new and rigorous quality criteria in the running of intercomparison exercises and the production and certification of RMs.

A recent intercomparison exercise for radionuclides in Mediterranean mussel, IAEA-437 [2], was organized by IAEA-MEL in response to the request received from CIESM in the framework of its Mediterranean Mussel Watch Program. Around 1080 kg of mussel (*Mytilus galloprovincialis* species) were collected in Anse de Carteau, Port Saint Louis du Rhône (43°20' S, 5°10' E), France, by the Institut de Radioprotection et de Sécurité Nucléaire (IRSN, France) in June 2003. The soft part of the sample was freeze-dried (0.4 dry/wet mass ratio), ground, sieved to 200 µm, then homogenized and bottled in 200 g aliquots under a nitrogen atmosphere. Preparation, storage and conservation for long shelf-life were carried out according to ISO recommendations. Homogeneity tests [2] indicated that the material is homogeneous for the suite of six alpha and beta/gamma emitters and for the range of weights tested. One sample of 200 g was distributed to each of 35 laboratories in November 2004. The exercise was expanded to a world-wide coverage, beyond the group of 15 laboratories participating in the Mediterranean Mussel Watch Program, in order to increase the likelihood of reaching the minimum number of results required for deriving recommended values for an intercomparison material. This was particularly required given the low levels of anthropogenic radionuclides in the sample. The deadline for reporting was extended to allow time for more reports to be submitted and the exercise was finally closed in March 2006, with a total response rate of 68%, respectively 47% for the CIESM Mussel Watch participating laboratories. The data, consisting in massic activities (Bq kg<sup>-1</sup> dry) decay-corrected to the reference date 1 November 2003, reported for 17 natural and 8 anthropogenic radionuclides, were treated according to the standard procedure applied at IAEA-MEL, described in [3] and references therein. Following the IUPAC and ISO recommendations for assessment of laboratory performance, the Z-score methodology was used for the evaluation of results. A preliminary certification of IAEA-385 was carried out according to the ISO Guide 35, the certification as RM remaining to be completed when more data from expert laboratories participating in the certification procedure will be available. For data sets comprising 5 or more accepted laboratory means, median values and confidence intervals (95% significance level) were calculated as estimations of true massic activities. The median values of the data within the confidence interval were considered as the recommended values when:

1. At least 5 laboratory means were available, calculated from at least 3 different laboratories.
2. The relative uncertainty of the median did not exceed ± 5% for activities higher than 100 Bq kg<sup>-1</sup>, ± 10% for activities from 1-100 Bq kg<sup>-1</sup> and ± 20% for activities lower than 1 Bq kg<sup>-1</sup>.

An activity value was classified as an information value when at least 5 laboratory means calculated from the results of at least 2 different laboratories were available. The median activities for the sets of accepted values were chosen as the most reliable estimates of the true values and were

given as recommended and information values. A summary of the preliminary values with confidence intervals for the most frequently reported anthropogenic and natural radionuclides is given in Table 1.

Tab. 1. Preliminary results of the IAEA-437 intercomparison exercise, designed to determine anthropogenic and natural radionuclides in a mussel sample from the Mediterranean Sea. The data received from 24 laboratories were evaluated. All the values are given for the reference date 1<sup>st</sup> November 2003 and expressed in Bq kg<sup>-1</sup> dry weight. <sup>210</sup>Po data are still under evaluation, therefore they were not included here. \*The values should be corrected for in-growth from <sup>241</sup>Pu.

Radionuclide	Median	Confidence interval (α = 0.05)
<u>Recommended value</u>		
<sup>40</sup> K	381	352 - 405
<sup>234</sup> U	2.34	2.20 - 2.44
<sup>239+240</sup> Pu	0.0076	0.0071 - 0.0087
<u>Information value</u>		
<sup>137</sup> Cs	0.18	0.07 - 0.31
<sup>226</sup> Ra	0.28	0.11 - 0.31
<sup>228</sup> Th	0.8	0.3 - 2.0
<sup>232</sup> Th	0.09	0.08 - 0.16
<sup>235</sup> U	0.08	0.03 - 0.12
<sup>238</sup> U	1.9	1.70 - 2.02
<sup>241</sup> Am*	0.006	0.004 - 0.022

This material provides an adequate test to laboratories confronted with reporting radioactivity data on Mediterranean marine samples. In this mussel sample anthropogenic radionuclides are for many laboratories near the limit of detection of combined radiochemical and (especially) radiometric methods. This is the case in particular for <sup>137</sup>Cs, the certification of which will involve measurements in underground laboratories, equipped beyond the technical capabilities of usual laboratories. Similarly, transuranics would be certified including reports from laboratories using mass spectrometric techniques. CIESM Mussel Watch laboratories could consider adopting periodic intercomparison runs and proficiency tests using a set of materials covering both low, typical Mediterranean, activities and medium activities, above the limit of detection of all the laboratories participating. This approach will allow a more comprehensive assessment of laboratory performance. At the same time, the results from this first intercomparison point to the capacity building needs necessary to address through national and international programmes in case the Mussel Watch Program is planned to continue running in the long term. The RM certification of IAEA-437 is expected for 2007.

### References

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