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Certified reference material for radionuclides in fish flesh sample IAEA-414 (mixed fish from the Irish Sea and North Sea)

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Abstract

A certified reference material (CRM) for radionuclides in fish sample IAEA-414 (mixed fish from the Irish Sea and North Seas) is described and the results of the certification process are presented. Nine radionuclides (⁴⁰K, ¹³⁷Cs, ²³²Th, ²³⁴U, ²³⁵U, ²³⁸U, ²³⁸Pu, ²³⁹⁺²⁴⁰Pu and ²⁴¹Am) were certified for this material. Information on massic activities with 95% confidence intervals is given for six other radionuclides (⁹⁰Sr, ²¹⁰Pb(²¹⁰Po), ²²⁶Ra, ²³⁹Pu, ²⁴⁰Pu ²⁴¹Pu). Less frequently reported radionuclides (⁹⁹Tc, ¹²⁹I, ²²⁸Th, ²³⁰Th and ²³⁷Np) and information on some activity and mass ratios are also included. The CRM can be used for quality assurance/quality control of the analysis of radionuclides in fish sample, for the development and validation of analytical methods and for training purposes. The material is available from IAEA, Vienna, in 100 g units. © 2006 Elsevier Ltd. All rights reserved.

Keywords: Certified reference material; Fish; Radionuclides

1. Introduction

Accurate and precise determination of radionuclide concentrations in marine samples is important for marine radioactivity assessments and for the use of radionuclides in the study of oceanographic processes. To address the problem of data quality, the IAEA Marine Environment Laboratory (IAEA-MEL) in Monaco has conducted intercomparison exercises on radionuclides in marine samples for many years as part of its contribution to the IAEAs programme of analytical quality control service (AQCS) (Sanchez-Cabeza et al., 2006; Povinec and Pham, 2001). An important part of this activity was the production of reference materials (RMs), which were usually products of worldwide intercomparison exercises. Regional intercomparison exercises generally resulted in RMs with only information values on radionuclide concentrations.

RMs are samples with well-established properties used for the assessment of analytical methods. Certified reference materials (CRMs) or standard reference materials (issued by the National Institute of Standard and Technology—NIST) (Inn et al., 1998) have property values certified by technically validated procedures (at least two independent analytical methods used) traceable to SI units (ISO, 1996, 2004). RMs and CRMs are valuable for method development and validation: they can indicate the need to improve or change existing methods and/or the need of further training. In fact, reference methods should only be accepted on the basis of interlaboratory tests performed on selected CRMs.

The IAEAs AQCS programme for radionuclides in the marine environment has recently focused on the production of CRMs, to improve the accuracy and precision of analyses carried out by the laboratories and thus the quality of data, and to provide traceability to SI standards. CRMs should be available for all important marine matrices, such as sediment, biota, sea water, suspended matter, etc.

This work was performed on a sample with enhanced radionuclide levels due to discharges from the Sellafield nuclear reprocessing plant. Participating laboratories were requested to determine as many anthropogenic radionuclides as possible by gamma spectrometry and other radionuclides requiring radiochemical separation (notably transuranics) by alpha spectrometry, beta counting and/or mass spectrometry. The certification process was completed and the material was issued as a CRM for radionuclides in fish flesh.

2. Methods

2.1. Description of the material

About 350 kg of mixed fish species were collected in the eastern Irish Sea by the Center for Environment, Fisheries and Aquaculture Science (CEFAS), Lowestoft, UK, in 1996. The skin was removed, the fish filleted and freezedried and subsequently sent to IAEA-MEL for processing. The sample, which was reduced by freeze-drying to about 69 kg, was then ground to powder, sieved through a 0.5 mm mesh and homogenized by mixing in a nitrogen atmosphere. The sample was freeze-dried once more, ground and sieved at 250 µm to obtain a final amount of 10 kg. As the quantity of material obtained was small and the measured activities of some anthropogenic radionuclides were relatively high, the sample was mixed with 100 kg of North Sea fish powder (fraction below 250 µm) to get the required quantity. To ensure homogenization of the final sample, both samples (fractions $<250 \,\mu\text{m}$) were mixed by jet pulverizing in a nitrogen atmosphere. The samples were then packed into brown glass bottles under nitrogen gas, sealed with polyethylene caps and labeled with the code IAEA-414. The sealed glass bottles containing 100 g of fish powder were sterilized according to ISO standards (ISO, 2004) at 10 kGy in an irradiation facility. The average moisture content of the lyophilized sample after bottling, determined by drying several aliquots in an oven at 80 °C to constant weight (1-2 days), was found to be approximately 3%.

Sample homogeneity was checked by measuring the activity of ⁴⁰K, ¹³⁷Cs, ²¹⁰Po, ²³⁵U, ²³⁸U, ²³⁸Pu, ²³⁹⁺²⁴⁰Pu and ²⁴¹Am of 16–30 bottles randomly chosen. Gamma spectrometry measurements were performed on 30–100 g

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