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Intercalibration Exercise for Difficult-To-Measure Radionuclides in Activated Steel

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ABSTRACT

Analysis of alpha and beta, namely Difficult-To-Measure (DTM), radionuclides in decommissioning waste is a challenging task since the DTM need to be separated radiochemically from the solid matrix and purified from each other and interfering radionuclides. In general, validation of the radiochemical method can be carried out by analysing a reference material. However, currently there are no commercially available reference material for DTM in decommissioning waste. Therefore, an international project, namely DTM-Decom, to carry out an intercomparison exercise on DTM analysis was developed within the NKS community. The aim of the DTM-Decom project in 2019 is to carry out an intercomparison exercise on analysis of DTM in activated steel. VTT will coordinate the project and provide the activated steel samples. All partners will analyse ^{55}Fe and ^{63}Ni and additionally some partners will also analyse ^{14}C and ^{59}Ni. The results will be published by the end of year 2019.

1 INTRODUCTION

Increasing number of research and power reactors are been shut down and decommissioned worldwide and within the Nordic countries. The operators are challenged with characterisation and categorisation of the decommissioning waste according to national demands in disposal facilities. Non-destructive techniques are available for determination of gamma emitting radionuclides whereas destructive analysis is needed for DTM radionuclides. The destructive analysis of DTM require radiochemical separation and purification methods, which often have several steps and depending on the material, the interfering radionuclides may cause significant uncertainties in the results. For example, analysis of ^{55}Fe and ^{63}Ni in activated steel are heavily interfered with ^{60}Co. Therefore, a validation of the measurement method is compulsory in order to produce reliable results. In analytical chemistry, reference materials are used for the validation. Radioactive reference materials are also available for analysis of radionuclides in environmental samples whereas there are no reference materials available for DTM analysis in decommissioning waste. One way of validating the radiochemical method is to use spiked samples which are often valid for testing, but it has been shown for example in a previous NKS intercalibration exercise [1] that spiked samples can behave very differently to real samples. This paper focuses on validation of the radiochemical method via carrying out an intercomparison exercise with several partners. The activated steel material was chosen to be the first material to be studied since all research and power reactors have steel in their waste inventory. Second material of interest for year 2020 is planned to be concrete. The project partners are VTT (FI), Helsinki University (FI), Fortum Power and Heath (FI), Cyclife (SWE), Technical University of Denmark (DK), IFE-Kjeller (NO), IFE-Halden (NO) and CEA (FR).

2 INTERCOMPARISON EXCERCISE

Discussions with the partners concluded that the DTM of interest in the activated steel samples will be ^{55}Fe and ^{63}Ni. Additionally, some partners will also analyse ^{14}C and ^{59}Ni. The results will be presented as scaling factors using ^{60}Co results provided by VTT. The intercomparison exercise will be carried out in the spirit of ISO 13528 international standard [2].

2.1 Sample selection and preparation

VTT has a reservoir of reactor pressure vessel (RPV) steel rods which have been irradiated in power reactors a few decades ago. A selection of the studied material was made based on having measurable amounts ^{55}Fe (t½=2.7 y) and ^{63}Ni (t½=100.1 y) in the samples while keeping the dose rate to maximum of 30 µSv/h. Thin 10 x 10 mm samples from RPV steel rods were cut using an electron discharge machine (EDM). The oxidation layer was removed by dipping the samples into oxidation removal solution and ethanol before letting them air dry. The samples were weight and placed individually in small plastic bags.

2.2 Homogeneity of the samples

Homogeneity of the samples was verified using ISOCS gammaspectrometry with an HPGe Be2020 spectrometer. All samples were individually measured using a sample holder with a constant distance from the detector in order to minimise double coincidence. The results showed that the samples were homogenous and contained ^{60}Co.

2.3 Sending of the samples

The samples were packed into small lead shields and carefully placed into cardboard boxes in order to minimise displacement during transport. The dose rates of the boxes were measured at surface and in 1 m of highest point of dose rate. Sending of the samples was carried out as an Excepted Package UN2910 transport. Each partner received 2-3 samples.

2.4 Radiochemical analysis

Each partner will carry out the DTM analysis using their internal radiochemical procedures. Even though detailed procedures may not be shared, the overall processes rely on complete destruction of the matrix using strong acids and separation and purification of ^{55}Fe and ^{63}Ni using ion exchange and chromatographic resins. Analytical techniques such as ICP-OES, ICP-MS and UV-Vis will be used for the determination of analytical yields while the analysis of DTM will be carried out mainly using LSC, but also gamma spectrometry for ^{59}Ni.

2.5 Analysis of the results

The collection and analysis of the results will be carried out during autumn 2019. The results will be decay corrected to a reference date and formation of a scaling factor using ^{60}Co results.

3 CONCLUSIONS

The intercomparison exercise to analyse DTM in activated steel will give validity for the radiochemical methods of the participating laboratories. The results will be published by the end of 2019.

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