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Production and characterization of a traceable NORM material and its use in proficiency testing of gamma-ray spectrometry laboratories

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HIGHLIGHTS

- A traceably characterized NORM material was produced and studied.
- The NORM material is made of quartz sand from a drinking water production site.
- A proficiency test with the new material was conducted.
- The samples were measured using high-resolution gamma-ray spectrometry.
- The paper outlines common problems with regard to proficiency tests of NORM.

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ABSTRACT

This paper outlines the process of characterizing a new NORM material for proficiency testing made of quartz sand with significantly elevated levels of ^{226}Ra obtained from the backflush of a drinking water treatment facility. Samples of the fully characterized NORM material were sent to European laboratories concerned with radioactivity measurements and environmental monitoring by gamma-ray spectrometry for proficiency testing. The paper discusses the results, specific requirements, problems and solutions that were found during the characterization process and the proficiency test.

1. Introduction

Naturally occurring radionuclides such as ^{40}K and the decay products of the primordial radionuclides ^{232}Th , ^{235}U and ^{238}U are present in many natural resources. Those radionuclides are responsible for the ever present background radiation that results in radiation doses of a few millisieverts per year (UNSCEAR, 2010). (Technologically enhanced) Naturally occurring radioactive materials ((TE)NORM) containing these radionuclides are exploited by industrial endeavours or used as building materials and often exceed the exemption limits of the activity concentration for radionuclides of the U and Th series, depending on mineral composition and geological origin (Maringer et al., 2010). Those industrial and public activities are generating a significant portion of NORM waste and can enhance the potential of radiation exposure of workers and the public, therefore, necessitating a careful

determination of activity concentration. Correct quantification of activity concentration and environmental monitoring require high methodical and metrological standards in the respective laboratories (Maringer, 2010). In order to correctly quantify the activity concentration in a sample with unknown activity it is necessary to perform a robust efficiency calibration. This can of course be done mathematically, but exact knowledge of one's measuring system is required as well as the exact sample composition, both of which are often unavailable. Another option is the use of traceable certified reference materials. After the detection system has been calibrated using the reference material, the activity of the unknown sample can be determined through comparison with the reference material. This is only valid, if the sample and the calibration source are similar, or, ideally, of the same composition and geometry (Gilmore, 2008).

There are basically two different kinds of reference materials in

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existence. Firstly, reference materials that have a simplified matrix, like pure water, and are artificially spiked with a known activity, and secondly, reference materials made from real sample material with a complex matrix depending on the origin of the material that is either naturally rich in radioactive substances or activity concentrations are enhanced by technological endeavours, but can also be spiked. While, on one hand, traceability can usually be ensured easily for the spiked materials, that process can be quite difficult for the naturally occurring materials. On the other hand, reference materials with a simple matrix are often very different from the real samples in terms of composition and density, impeding accurate evaluation of their activity concentration by comparison with the reference materials (Larijani et al., 2017). To simply transfer the efficiency calibration obtained with a simplified matrix can – depending on the composition of the unknown sample – lead to a wrong estimation of activity concentration, due to neglect of factors like the self-absorption of the material. This, in turn, can lead to potentially dangerous situations or entail expensive repercussions because decisions concerning radiation protection, waste disposal, etc. were made based on erroneous measurement results. For the traceable determination of activity concentrations in (TE)NORM, reference materials with a well-established activity concentration as well as a realistic sample matrix are essential.

In Austria and many other countries, accredited laboratories concerned with gamma-ray spectrometric measurements are obligated to participate in intercomparison exercises or proficiency tests in order to prove their competence in the field and prove periodically conformity with the quality and competence requirements of the ISO 17025 standard. In the past, BEV has organised a number of comparison exercises with matrices spiked with ^{57}Co , ^{60}Co and ^{137}Cs and while proficiency in general was high for these “standard radionuclides”, the evaluation of activity concentrations for samples with densities $\approx 1 \text{ g/cm}^3$ and radionuclides of the natural decay chain is much more complex. Many of the radionuclides of the natural decay chains have a number of different emission lines, most with very low emission probabilities, or cannot be directly evaluated using gamma-ray spectrometry. This requires long measurement times and a low background environment, and other factors, like sample equilibrium, have to be considered. Additionally, many of the lines show significant interference with the lines of other nuclides which makes the evaluation of the activity concentration much more complex (Baumgartner et al., 2017) and suitable reference materials are needed.

For this study, a NORM material for proficiency testing was made of quartz sand taken from a drinking water treatment facility in Lower Austria. For the characterization process, methods developed in the European Metrology Joint Research Project EMRP IND57 MetroNORM “Metrology for processing materials with high natural radioactivity” (Maringer et al., 2017) were used. MetroNORM focused on creating traceable, accurate, and standardized measurement methods, reference materials and systems for application in the concerned industries. The NORM material was sent to various laboratories concerned with radioactivity measurements and environmental monitoring, where the activity concentration for ^{40}K , ^{226}Ra and ^{228}Ra was evaluated in the respective laboratory’s customary measurement geometry.

2. Gamma-ray spectrometry detector setup for characterization of the NORM material at BEV

Measurements were conducted with a Canberra Industries HPGe GC3020 p-type standard electrode coaxial germanium detector (crystal diameter: 56 mm, crystal height 54 mm; rel. eff.: 26.8%, peak to Compton ratio: 58:1; FWHM: 1770 eV at 1332 keV, 834 eV at 122 keV) inside an aluminium end cap and equipped with a lead shielding (100 mm Pb, 1 mm Cd, 1 mm Cu) for verifying homogeneity of the final samples as well as for establishing the reference values. The spectra were analysed with Canberra’s Genie2000 software. The detector system is traceably calibrated using standard solutions standardized

with the Austrian national standard for activity, an ISOCAL IV ionization chamber traceably calibrated at the National Physical Laboratory (NPL), UK and traceably calibrated reference point sources manufactured by Physikalisches-Technische Bundesanstalt (PTB).

3. Origin of the material

The material for the proficiency test has been taken from the backflush of a drinking water treatment facility in Lower Austria, in a region where the water contains significantly elevated levels of ^{226}Ra due to the underlying geological conditions. Magmatite, granite and clastic sediments such as clays, gravel, and sand predominate. The water is rich in iron and manganese. At the treatment facility, drinking water is oxygenated, causing iron and manganese (usually in the form of hydrogen carbonates) to oxidize. The water is then passed through a bed of quartz sand acting as a filter where the hydroxides of iron and manganese, as well as of radium, precipitate. A filter holding back the sand closes off the system. Once a month, this filter is cleaned by flushing water in the reverse direction. The result of this water treatment process is a quartz sand rich in ^{226}Ra that has been used as a material in this study.

4. Sample treatment and homogeneity

The original material taken from the drinking water production plant was roughly homogenized, dried at 105 °C and sifted by passing it over screens of varying size to remove any other material. After drying again for a week at 105 °C, the sand was carefully homogenized using a three-dimensional shaker mixer WAB Turbula T2F.

Preliminary gamma-spectrometric measurements were performed in order to check for homogeneity issues in the quartz sand. This was done by measuring count rates for the 46.5 keV line of ^{210}Pb , 1460.8 keV line of ^{40}K , 186.2 keV line of ^{226}Ra as well as the 583.4 keV and 911.2 keV lines of ^{228}Ac for different grain size fractions. The results showed that the samples were not homogeneous and further homogenisation steps were necessary. Upon further testing it was concluded that the very fine grain size portion (< 0.56 mm) causes problems with homogeneity. Preliminary measurements showed that the activity concentration in the very fine-grained fraction (< 0.56 mm) of the sample material is much higher (see Table 1).

Therefore, only material with a grain size between 0.8 mm and 2 mm was used to prepare the samples. 200.00 g \pm 1.00 g portions of the grain size fraction of (0.8–2) mm were filled into cylindrical polypropylene sample containers ($r = 3.4 \text{ cm}$, $h = 7.7 \text{ cm}$) to produce the samples for the proficiency test.

The homogeneity of the sample collective was studied only between containers, not within containers, by comparison of the count rates of the aforementioned lines. Measurement times for the homogeneity testing varied between 7 h and 12 days, depending on laboratory use. Fig. 1 gives the weighted mean of the relative deviations of all considered energy lines (see above) of the respective radionuclide for each sample. The final uncertainties of the respective sums of relative deviations are calculated using Gaussian propagation of uncertainties.

According to the results of the homogeneity testing of the samples of grain size fraction 0.8–2 mm, the samples 9, 28, 30, 31 and 33 were deviating from the mean at an unacceptable level and eliminated. The eliminated samples show a trend to higher numbers and, therefore,

Table 1
Comparison of activity concentration of two grain size fractions of one test sample.

Grain size fraction	^{226}Ra (Bq/kg)	u_{rel} (%)	^{228}Ra (Bq/kg)	u_{rel} (%)	^{210}Pb (Bq/kg)	u_{rel} (%)
0.8–2 mm	999	23	1080	5	131	6
< 0.56 mm	8660	6	7670	5	710	6

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