Development of innovative methods in radionuclide metrology for applications in natural resources and life sciences

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Abstract. The main problem with naturally occurring radioactive materials lies in the wide variety of chemical elements and radionuclides composition. The naturally occurring radionuclides in these materials emit many gamma-rays of different, and sometimes interfering, energies that must be measured and analyzed by an expert. For the traceable determination of activity concentrations, reference materials with a well-established activity concentration are essential. Therefore, an extensive study of particular problems arising in gamma-ray spectrometry of naturally occurring radioactive materials has been conducted to investigate the best gamma-lines to analyze the activity concentration. A novel alternative way to approach this problem is the use of artificial neural networks. Their use serves as a new calibration where no expert knowledge of gamma-ray spectrometry is needed by the end-user. In this work, an artificial neural network was created that can decide from the input data of a raw gamma-ray spectrum, if the activity concentrations in a sample are above or below the exemption limits. Additionally, a certified traceable reference material has been established. It is made from filter sand of a drinking water production site with significantly elevated levels of ^{226}Ra and was used in an intercomparison exercise with nine European laboratories concerned with radioactivity measurements. Due to its volatile nature, special attention is given to the evaluation of radon activity concentration and radon tightness of used sample containers. A simple and sensitive method for the estimation of ^{222}Rn leakage is proposed. Furthermore, two ionization chamber methods have been used to evaluate the radon activity concentration in drinking water samples. A short overview of the results of these studies, the preparation of the reference material and the results of the intercomparison exercise and artificial neural networks will be presented in this paper.

KEYWORDS: radionuclide metrology, gamma-ray spectrometry, NORM, traceably reference material, artificial neural networks, radon in water

1 INTRODUCTION

Naturally occurring radionuclides like ^{40}K and the decay products of the primordial radionuclides ^{232}Th, ^{235}U and ^{238}U are present in many natural resources. Naturally occurring radioactive materials (NORM) containing these radionuclides are exploited industrially and can be found in some of the most important industries, such as drinking water production, oil/gas production, coal industry, metallurgy, rare earth industry, phosphate industry, production of TiO_2, etc. and are often used as building materials (e.g. [1], [2], [3], [4]). These industrial activities generate a significant portion of waste, possibly enhancing the potential of exposure of workers and the public [5]. Accurate assessment of activity concentrations of various radionuclides present in NORM is paramount as a basis for waste disposal, possible re-use of materials and ensuring radiation protection of workers, the public and the environment. Since the raw materials and processed substances within the NORM industries display a huge diversity regarding their chemical composition and physical nature, evaluation of activity concentration is often not straight-forward and requires time-consuming and laborious deconvolution of gamma-spectra by experts. To accomplish these objectives, traceable, accurate and standardized measurement techniques and instruments are needed, particularly for in-situ applications [5].

2 STUDY OF PARTICULAR PROBLEMS ARISING IN GAMMA-RAY SPECTROMETRY OF NORM SAMPLES

2.1 Overview

Activity concentrations of NORM samples are usually evaluated using low-level gamma-ray spectrometry as it is a versatile and non-destructive method with which several radionuclides can be evaluated at the same time [3]. However, a few particular problems arise in the analysis of activity concentration of NORM samples which require expert knowledge.

Due to the presence of ^{232}Th, ^{235}U, ^{238}U and their progeny, there are many different gamma-lines present in NORM spectra. Many of the gamma-lines have (very) low emission probabilities, are subject to true coincidence summing and a large number of spectral interferences occur between the many nuclides of the natural decay series [1]. Resolution of such peaks is usually difficult, if not impossible. Activity concentration in environmental samples is usually low and often does not significantly exceed the background. This leads to long measurement times in order to obtain significant results with sufficiently low uncertainties, especially if it is not possible to use specialized low-level detectors. Additionally, the detector background is often comprised of radionuclides of the natural decay chains that surround the detector and can emanate from building materials, which in turn interfere with the sample spectrum. Moreover, radioactive equilibrium may also be disturbed due to natural events or processing of the material ([2]; [5]), making it impossible to use the activity concentration of the daughter nuclides to establish activity concentration for parents that have no or unsuitable gamma-lines. Furthermore, loss of radon gas from the sample container can lead to (significant) underestimations of activity concentrations for ^{226}Ra and its mother nuclides if radon progenies are used for the evaluation. Determination of (the degree of) radon tightness for all used sample containers is, therefore, paramount to ensure safe and sound decision making when it comes to radiation protection issues.

2.2 Investigated NORM samples

Exemplary for spectra in which interferences occur, the following materials were selected for a detailed investigation: tuff (volcanic ash, a common building material), titanium dioxide waste, ion exchange resin (from a waterworks purification filter) and a filter residue (origin unknown). For these materials, the main sources of spectral interferences were evaluated for selected gamma-emitting radionuclides of the natural decay series and ^{40}K.

2.3 Nuclear data and calculation of deviation

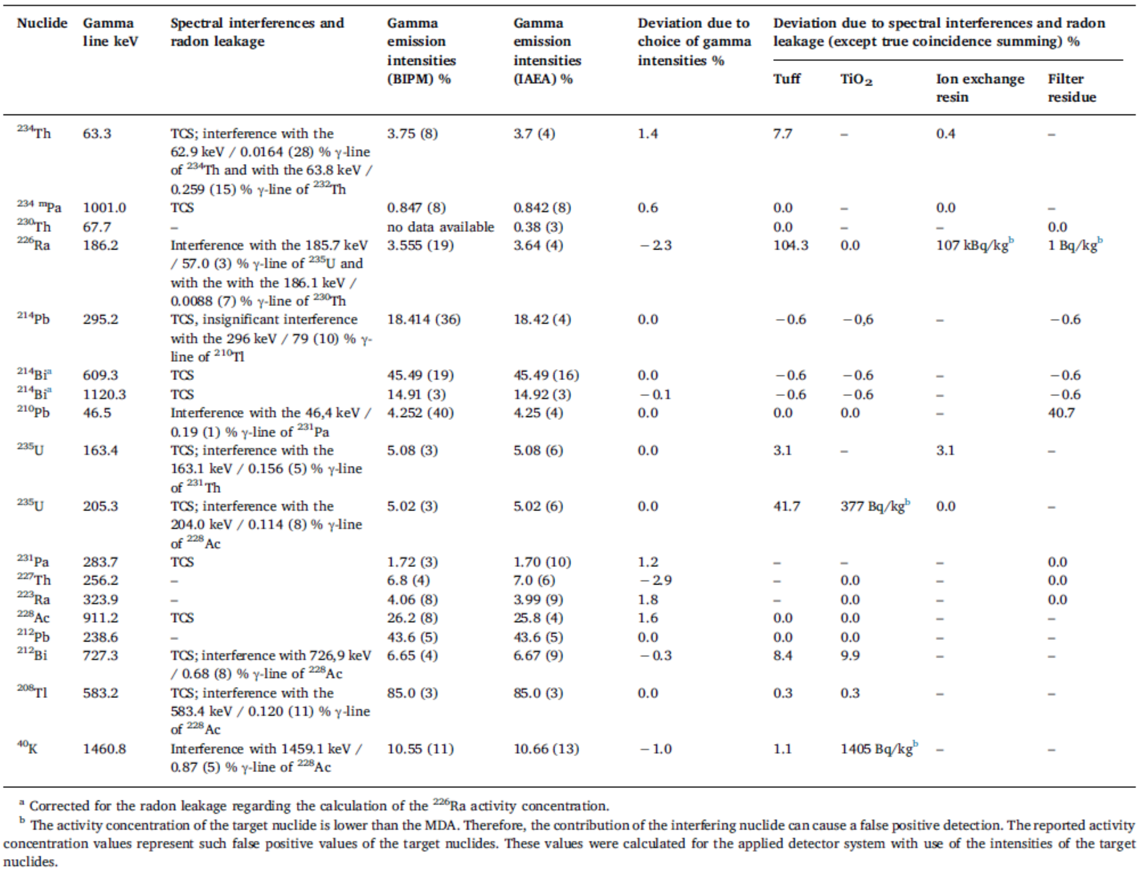
All decay data in this section were taken from BIPM [6], except the column "Gamma emission intensities (IAEA)" in Table 1 and the data for 230Th are from IAEA [7]. For 230Th there are no data available from BIPM. Other literature sources also give comprehensive recommendations on gamma-lines and their interferences (e.g [8]). Specifically, interferences in NORM samples were studied, e.g. in [5], [9]).

For the selected materials, a critical peak-by-peak evaluation on spectral interferences of selected gamma-emitting radionuclides of the natural decay series and ^{40}K was performed. The deviation due to spectral interferences is calculated as the ratio of the count rates of the interfering photons and the photons of the nuclide of interest. This means that neglecting the interference would cause the deviations given in Table 1 (e.g. if the peak in the spectrum at 1460.8 keV is generated equally by ^{40}K and ^{228}Ac the value for the deviation would be 100 %).

2.4 Radon tightness of sample containers

The gamma spectrometric measurement of ^{226}Ra can be done either by a direct measurement of the 186 keV gamma-line or by measuring the gamma-lines of the ^{222}Rn progenies ^{214}Bi and ^{214}Pb. Due to an interfering gamma-line, a direct measurement requires the deconvolution of the full energy peak [10] and the exact knowledge of the ^{235}U activity concentration, see e.g. [11]. The more common method of determining ^{214}Bi and ^{214}Pb strongly relies on the radon tightness of the sample containers [12], [13]. For the gamma spectrometric measurements, cylindrical polystyrene containers were used. The samples were sealed with glue and stored before the measurements to ensure secular equilibrium between ^{226}Ra and the ^{222}Rn progenies ^{214}Bi and ^{214}Pb. Besides the ^{222}Rn leakage, the distribution in solid/liquid and air phases in gamma-ray spectrometry must be considered for partially filled sample containers [14]. This paper proposes a simple method for the assessment of the ^{222}Rn leakage of sample containers. The method is based upon the measurement of the ^{222}Rn activity concentration inside a 3 L desiccator. The sample and a Canary Pro ^{222}Rn in air measuring device are placed inside the desiccator and the ^{22}Rn build-up curve is measured. The measurement results show a loss of radon gas of approximately 0.6 % for the completely filled polystyrene container and 0.7 % for the half-filled liquid sample. Since the liquid sample has a large void volume and a low radon retention, it gives an estimate for a high exhalation of a sample.

Table 1: Evaluation of particular gamma-ray spectrometry problems on the basis of selected NORM samples including the calculation of interference contributions, a comparison of BIPM and IAEA gamma-emission intensities for selected natural radionuclides as well as radon leakage effects.



Nuclide	Gamma line keV	Spectral interferences and radon leakage	Gamma emission intensities (BIPM) %	Gamma emission intensities (IAEA) %	Deviation due to choice of gamma intensities %	Deviation due to spectral interferences and radon leakage (except true coincidence summing) %			
						Tuff	TiO_2	Ion exchange resin	Filter residue
^{234}Th	63.3	TCS; interference with the 62.9 keV / 0.0164 (28) % γ-line of ^{234}Th and with the 63.8 keV / 0.259 (15) % γ-line of ^{232}Th	3.75 (8)	3.7 (4)	1.4	7.7	–	0.4	–
^{234m}Pa	1001.0	TCS	0.847 (8)	0.842 (8)	0.6	0.0	–	0.0	–
^{230}Th	67.7	–	no data available	0.38 (3)		0.0	–	–	0.0
^{226}Ra	186.2	Interference with the 185.7 keV / 57.0 (3) % γ-line of ^{235}U and with the with the 186.1 keV / 0.0088 (7) % γ-line of ^{230}Th	3.555 (19)	3.64 (4)	−2.3	104.3	0.0	107 kBq/kg[b]	1 Bq/kg[b]
^{214}Pb	295.2	TCS, insignificant interference with the 296 keV / 79 (10) % γ-line of ^{210}Tl	18.414 (36)	18.42 (4)	0.0	−0.6	−0,6	–	−0.6
^{214}Bi[a]	609.3	TCS	45.49 (19)	45.49 (16)	0.0	−0.6	−0.6	–	−0.6
^{214}Bi[a]	1120.3	TCS	14.91 (3)	14.92 (3)	−0.1	−0.6	−0.6	–	−0.6
^{210}Pb	46.5	Interference with the 46,4 keV / 0.19 (1) % γ-line of ^{231}Pa	4.252 (40)	4.25 (4)	0.0	0.0	0.0	–	40.7
^{235}U	163.4	TCS; interference with the 163.1 keV / 0.156 (5) % γ-line of ^{231}Th	5.08 (3)	5.08 (6)	0.0	3.1	–	3.1	–
^{235}U	205.3	TCS; interference with the 204.0 keV / 0.114 (8) % γ-line of ^{228}Ac	5.02 (3)	5.02 (6)	0.0	41.7	377 Bq/kg[b]	0.0	–
^{231}Pa	283.7	TCS	1.72 (3)	1.70 (10)	1.2	–	–	–	0.0
^{227}Th	256.2	–	6.8 (4)	7.0 (6)	−2.9	–	0.0	–	0.0
^{223}Ra	323.9	–	4.06 (8)	3.99 (9)	1.8	–	0.0	–	0.0
^{228}Ac	911.2	TCS	26.2 (8)	25.8 (4)	1.6	0.0	0.0	–	–
^{212}Pb	238.6	–	43.6 (5)	43.6 (5)	0.0	0.0	0.0	–	–
^{212}Bi	727.3	TCS; interference with 726,9 keV / 0.68 (8) % γ-line of ^{228}Ac	6.65 (4)	6.67 (9)	−0.3	8.4	9.9	–	–
^{208}Tl	583.2	TCS; interference with the 583.4 keV / 0.120 (11) % γ-line of ^{228}Ac	85.0 (3)	85.0 (3)	0.0	0.3	0.3	–	–
^{40}K	1460.8	Interference with 1459.1 keV / 0.87 (5) % γ-line of ^{228}Ac	10.55 (11)	10.66 (13)	−1.0	1.1	1405 Bq/kg[b]	–	–

[a] Corrected for the radon leakage regarding the calculation of the ^{226}Ra activity concentration.

[b] The activity concentration of the target nuclide is lower than the MDA. Therefore, the contribution of the interfering nuclide can cause a false positive detection. The reported activity concentration values represent such false positive values of the target nuclides. These values were calculated for the applied detector system with use of the intensities of the target nuclides.

3 COMPARISON OF MEASUREMENT METHODS USED IN ANALYSIS OF DRINKING WATER SAMPLES

3.1 Overview

The greatest potential danger of radon in water is not its occurrence dissolved in drinking water, where concentrations are usually quite low, but the additional amount of radon released to the atmosphere [15], causing high dose rates when accumulation happens. The WHO estimates, that 90 % of the dose attributable to radon in drinking-water comes from inhalation rather than ingestion [16]. The aim of this exercise was to compare two different ionization chamber measurement methods for radon in drinking water and gain insights into the measurement process.

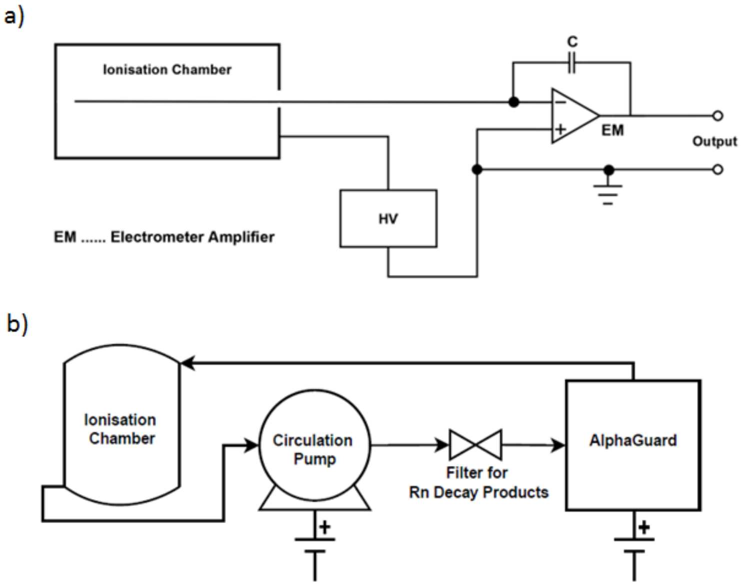
3.2 Sampling

The samples used in this study were mainly taken from springs along the so called “Thermenlinie”, a seismic disturbance zone west of Vienna running from the Northern Limestone Alps to the south of Vienna along the edge of the Vienna Basin. Commercially available glass water bottles have been used as sample containers. The bottles are cheap, easily available, clean with regard to radon measurement and sufficiently gas tight [17]. In order to retain as much radon in the water as possible and prevent early radon emanation from the water, the bottles were carefully and slowly filled using a hose to prevent bubbling water. The hose was inserted into the bottle, letting the water flow in at the bottom and slowly raising the water level. When full, the water was left running over the rim for a few seconds and then the hose was slowly retrieved. After decanting a small amount of water to create room for the developing gases, the bottle was sealed using plastic screw tops.

3.3 Measurement setup

The two measurement set-ups that were compared as part of this exercise are an AlphaGUARD radon monitor (BEV's national standard for radon activity concentration per volume unit), traceably calibrated at ENEA, and a self-built cylindrical ionization chamber with a volume of 9.3 L. Fig. 1 shows the measurement setups. The ionization chamber is supplied by 260 V and connected to an electrometer. Activity concentration is determined via a charge measurement. This way, electrical current is integrated over a period of time and fluctuations in electrical current have less influence on the result, therefore increasing precision. The ionization chamber's inner pressure was lowered significantly with a water jet pump connected to a tap. The sample container was attached directly to the ionization chamber by using a plug with two hoses. Slightly opening the valve at the chamber results in a flux of ambient air that is slowly sucked through the water and chamber. The flowing air causes the water to bubble and very efficiently emanates radon from the water. A previous study [18] showed that three times the volume of the liquid is enough to emanate and transfer virtually all the radon from the water into the chamber (chamber volume approx. 9 L vs. approx. 1 L water sample). Once the pressure in the chamber is balanced with the ambient air, equilibrium of radon and its daughters starts to build up. When measurements with the ionization chamber are finished, the outlets of the chamber are fitted with hoses and connected to the AlphaGUARD. A circulation pump creates a flux of 1 L/min. After ensuring that the air is circulated at least twice through the AlphaGUARD, the pump is shut off and the ionization chamber disconnected. Since secular equilibrium is reached after only about ten half-life periods of the longest-lived daughter, ^{222}Rn and its progeny would only be in equilibrium after approx. five hours (ten half-lives (26.8 (9) min) of ^{214}Pb).

Figure 1: Set-up of the measurement system: (a) Ionisation chamber, (b) AlphaGUARD.



3.4 Results

Results and uncertainties given by both measurement set-ups agree in general except for a few measurements (difference factor of 1.00 - 1.55). Relative uncertainties range from 10 % to 35 % ($k = 1$). The insights into the sampling and measurement process were used to design a new measurement method for radon in water which will be published in the future. More information on this study can be found in [19].

4 PRODUCTION AND CHARACTERIZATION OF A TRACEABLE NORM REFERENCE MATERIAL AND ITS USE IN PROFICIENCY TESTING

4.1 Overview

For laboratories that routinely analyze the same material in a standard geometry, the use of certified reference materials can be easier to use and cheaper in the long term compared to the use of Monte Carlo methods for efficiency calibration of detectors. Since there are many different requirements to be met for each application and laboratory, there is a general need for certified NORM reference materials in science and industry with sufficiently low uncertainties to achieve traceability, validate methods and calibrate instruments [20], [21], [22]. An important part of this work was the production and characterization of a non-spiked, as close to natural as possible reference material traceable to an international metrological standard of radioactivity measurement. The reference material was then used in proficiency testing of nine European laboratories tasked with radioactivity measurements.

4.2 Origin of the material

The material to produce the reference material 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 in this area and 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 the base material to produce the reference material in this study.

4.3 Material treatment

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. In order to obtain a stable reference material retaining as much of its natural matrix as possible, only the grain size fraction of (0.8 – 2.0) mm was used. The smaller grain size fractions proved to be too mobile, thus causing instability in the reference material, and were therefore removed. After drying again for a week at 105 °C, the sand was carefully homogenized using a three-dimensional shaker mixer WAB Turbula T2F. After ensuring homogeneity, the traceable reference values of the NORM radionuclides were established by BEV (the National Metrology Institute of Austria) as (0.605 ± 0.024) Bq/g ^{40}K, (1.003 ± 0.015) Bq/g ^{226}Ra, and (0.806 ± 0.013) Bq/g ^{228}Ra (105 ◦C dry weight basis, $k = 1$).

4.4 Proficiency testing

Samples of quartz sand that were deemed homogenous were sent to nine European laboratories concerned with radioactivity measurements and environmental monitoring. The laboratories received one to four samples of 200 g each, depending on their requested amount of material. According to the proficiency test protocol, the sample material was dried at 105 °C at the laboratories and then transferred into the respective laboratory's preferred measurement container where the activity concentrations of ^{40}K, ^{226}Ra and ^{228}Ra were determined on HPGe detectors according to the laboratories' quality management procedures.

4.5 Conclusions

For this study, a natural matrix, non-spiked NORM material made of quartz sand with elevated levels of radioactivity due to the treatment of drinking water was produced and used in a proficiency test exercise with nine European laboratories. Homogeneity of the samples was assessed by comparing count rates of strong emission lines. Five samples were excluded from the exercise as they were deviating too much from the mean, but most samples were deemed sufficiently homogeneous. Finely grinding the quartz sand would ensure better homogeneity but also result in a less “realistic” reference material since grinding would destroy the material's natural matrix. The results of the NORM proficiency test showed the expected behavior: larger deviations from the mean and reference value, and a larger general spreading of results for NORM than for simple matrices and anthropogenic radionuclides like ^{57}Co, ^{60}Co and ^{137}Cs and a general underestimation of the ^{226}Ra activity concentration. One reason for this outcome may be that approximately half the participating laboratories had only little experience in analyzing NORM radionuclides. While the results for ^{40}K and ^{228}Ra are generally satisfactory, it is clear from the results of this proficiency test that many laboratories have problems with the determination of the activity concentration of ^{226}Ra, specifically. One reason for the underestimation of the ^{226}Ra activity concentration of up to 35 % is the use of unsealed or not radon tight sample containers or measurement before reaching radioactive equilibrium. During the evaluation of the results, it also became evident that many participants had problems with the correct treatment of measurement uncertainties and conversion of quantities, especially when specifying parts and multiples of quantities. Furthermore, it is obvious that consistent and complete record keeping on sample treatment and activity concentration evaluation is imperative and not typically performed by most laboratories. This necessitates awareness building and training courses for the responsible personnel. One of the lessons learned in this proficiency test is that reporting sheets should be kept as short and simple as possible to ensure that participants will provide complete information and that only one language should be used as translations can produce some ambiguous phrasing. In general, the result of this proficiency test showed the potential for further improvements in correctly analyzing the activity concentration of natural radionuclides in NORM, even for expert laboratories. The detailed results were published in [23].

5 FEASIBILITY STUDY: ARTIFICIAL NEURAL NETWORKS FOR MONITORING OF NORM

5.1 Overview

As can be seen from the above chapters, the evaluation of activity concentration by gamma-ray spectrometry is a complicated process that requires expert knowledge. Furthermore, quick and reliable methods are needed to determine activity concentration in raw materials and waste, e.g. at industrial sites [19]. As part of this work a feasibility study [24] was undertaken to test if artificial neural networks (ANNs) can be used as a novel method to determine if NORM samples are above or below the exemption limit of 1 Bq/m3 (as laid down in Council Directive 59/2013/Euratom [25] from a raw gamma-spectrum, thus avoiding the complex analysis associated with spectral deconvolution.

5.2 ANNs

ANNs are mathematical software tools that can learn from training datasets and apply that “knowledge” to unknown problems. ANNs are very versatile and can handle imprecise and noisy information. Once the ANN is set up, no detector calibration or specialized end-user knowledge is required. The final ANN serves as an alternative detector calibration and can function like a black box for the end-user.

5.3 Training data

The quality of the ANN's results is determined by the representativeness of the used training data. In this case, equilibrium and disequilibrium situations have been studied for seven different NORM (ilmenite, sand, TiO_2, tuff 1, tuff 2, phosphogypsum MetroNORM, phosphogypsum Huelva) and a total of 635 training cases have been calculated using the Monte Carlo codes PENELOPE and PENNUC.

The training input consists of a (635 x 15) matrix, containing the count rates of twelve relatively interference free gamma-lines, as well as material density, a material identifier, sample mass, and an assigned activity category. The output (or “result”) is a set of 12 activity categories corresponding to each input gamma-line. Activity categories corresponding to the value of the exemption limit, a little above or below the exemption limit and far above or below the exemption limit were used as a quick method of analysis instead of Bq/g as a quick way of checking the results in this feasibility study.

5.4 Final ANN

The output of the final ANN is an activity category for each input gamma-line. The performance of the final ANN was investigated using real gamma-spectrometry data for each material. The ANN's output agrees very well with the manual evaluation. Training the ANN in that way resulted in an ANN with an overall regression factor of 0.9975. This shows that ANNs are well-suited for the task of deciding if a material is above or below the exemption limit from a raw gamma-spectrum.

5.5 Conclusions

This study was undertaken to find out if ANNs can be applied to the problem of determining if the activity concentration in a sample is above or below the exemption limit. It was shown that the ANN was able to correctly classify all the testing materials and ANNs are well-suited to carry out this task. The lack of real sample material has been sidestepped by calculating artificial spectra using Monte Carlo calculations which necessitate the complicated and time-consuming study of disequilibrium situations. For specialized industries where only one material is analyzed and many sample spectra are available as training input, the author proposes the use of an ANN specifically trained to only that purpose. The ANN functions as an alternative calibration method and no detailed detector knowledge is necessary, as long as all spectra come from the same source. In theory, an ANN designed in this way could function like a black box for an untrained worker, significantly reducing the need for highly trained and specialized personnel. For the end-user only a gamma spectrum in ASCII format and no specialized knowledge whatsoever in the field of gamma-ray spectrometry is required.

6 SUMMARY

A study of particular problems appearing in gamma-ray spectrometry of NORM samples showed that gamma-ray spectrometry of NORM samples is challenging and requires expert knowledge in spectrum deconvolution due to the many different radionuclides of the natural decay chains present within NORM samples. Spectral interferences were studied and a best practice recommendation on suitable gamma lines for the evaluation of activity concentration was published. Furthermore, it was found that the determination of ^{226}Ra based upon ^{214}Bi and ^{214}Pb is strongly dependent on the radon tightness of the sample containers. A simple method to assess leakage of radon gas from the sample containers was proposed. [1]

During the investigation it was found that gamma-ray spectrometry of NORM samples heavily relies on the availability of certified reference materials. The use of Monte Carlo codes for simulation of radiation transport can alleviate this problem but exact knowledge of the detector system and measured sample are required. Due to the large number of different NORM, there is a general need for more reference materials in industry and science. Therefore, a natural matrix, non-spiked NORM material made of quartz sand with elevated levels of radioactivity caused by the treatment of drinking water was produced. The consecutive use of the material in a proficiency test showed the expected behavior: larger deviations from the mean and reference value, and a larger general spreading of results for NORM than for simple matrices and anthropogenic radionuclides like ^{57}Co, ^{60}Co and ^{137}Cs and a general underestimation of the ^{226}Ra activity concentration. ^{226}Ra activity concentration was underestimated by up to 35 % ($k = 1$). This underestimation is caused by the use of sample containers that are either not sufficiently radon tight or measurement before reaching radioactive equilibrium. It was demonstrated that evaluation of ^{226}Ra is often not correctly performed which underlines the importance of trained gamma-ray spectrometry experts. [23]

To alleviate this problem and allow for quick decision making in the field, a feasibility study was undertaken to test if artificial neural networks can be applied to the problem of determining whether the activity concentration in a sample is above or below the exemption limit. An ANN with the ability to analyse a raw gamma-spectrum and determine if the activity concentration in 12 gamma-lines is above or below the exemption limits laid down in national legislation was created. The ANN was trained using artificially prepared spectra that were calculated using the Monte Carlo codes PENELOPE and PENNUC, and Matlab. The ANN's performance was tested using real gamma-spectra. The network was able to correctly classify all of the testing materials. Therefore, ANNs are well-suited to carry out this task. For the end-user, only a gamma spectrum in ASCII format and no specialized knowledge in the field of gamma-ray spectrometry is required. [24]

Furthermore, two ionization chamber measurement methods for the evaluation of radon activity concentration in drinking water have been compared [19].

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