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Advancements in NORM metrology – Results and impact of the European joint research project MetroNORM

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A R T I C L E I N F O

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A B S T R A C T

The results of the three years European Metrology Research Programme's (EMRP) joint research project 'Metrology for processing materials with high natural radioactivity' (MetroNORM) are presented. In this project, metrologically sound novel instruments and procedures for laboratory and in-situ NORM activity measurements have been developed. Additionally, standard reference materials and sources for traceable calibration and improved decay data of natural radionuclides have been established.

1. Introduction

Naturally occurring radionuclides are present in many natural resources and such as raw materials and mineral feedstocks (EC, 1999, 2003; OGP, 2016; WNA, 2016). Industrial activities that exploit these resources may lead to enhanced potential for exposure to Naturally Occurring Radioactive Materials (NORM) in products, by-products, residues, and wastes (Smith, 1992; EC, 2003, 2013, Kunze et al., 2007, Maringer et al., 2010). Industries working with NORM raw materials also produce large amounts of waste, and such waste materials constitute a huge economic and ecological burden if not properly disposed or re-used.

The European metrological joint research project MetroNORM addresses these issues by developing new traceable measurement capabilities for NORM industries in order to significantly improve the industrial processing of NORM resources and waste. Traceable, accurate, and standardised measurement methods and instruments, in particular for in-situ applications, are needed to decide on the re-use of waste materials without increasing costs whilst avoiding contamination of the environment and exposure of the public (Maringer, 2008, 2010). Ionising radiation measurement in the recycling industry currently generally focuses on artificial radionuclides (e.g. ^{60}Co,

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^{137}Cs). The developed measurement capabilities will include NORM measurement systems for use in the laboratory and on-site (i.e. in situ) and reference/calibration materials for validation of such measurement systems.

Due to the radioactivity content of NORM resources, there is a need to control the radiation exposure to workers and members of the public in accordance with current international radiation protection safety standards. For example, in commercially exploited rare earths deposits, the activity level of thorium and uranium, depending on the type of mineral and its region of occurrence, generally exceed the worldwide median values for soil by up to 200 times of thorium and up to 30 times in the case of uranium (IAEA, 2011, p. 10). When such minerals are being handled or processed, it is therefore vital to determine the NORMs present and their activity as reliable and accurately as possible. Thus, reference materials are needed to calibrate and validate the applied radioanalytical methods and instruments involved as well as improved analysis and interpretation of the results.

Further to this, traceable and accurate measurement methods and systems, in particular for in-situ use, are needed to accurately determine the re-use of waste NORM materials, in order to avoid contamination of the environment and over exposure to the public. Currently, ionising radionuclide metrology and measurements in the industry focus mainly on artificial radionuclides, and NORM radionuclides are commonly considered as part of the natural background, regardless of their concentration (Maringer et al., 2008a, Paiva et al., 2004; Pommé and Sibbens, 2008; Pommé et al., 2009a, 2009b; Ratel et al., 2008). Therefore, a reliable measurement of NORM radionuclides is needed.

The production processes in NORM industry facilities must be effective and safe, and hence the NORMs in the waste produced must be identified as well as accurately measured (Arinc et al., 2011; Bertolo et al., 2006; Carconi et al., 2012; Carrapiço et al., 2007; Dryak and Kovar, 2006; Ferreux et al., 2009; Korun et al., 2012a, 2012b; Lépy et al., 2012; Ruth and Burney, 2008). Therefore, improved decay data is needed for selected natural radionuclides such as the ^{238}U (uranium) and ^{235}U decay chains. Furthermore, to ensure accurate measurements, the measurement systems must be traceably calibrated with calibration standards that are relevant to the real composition and geometry of the NORM materials being measured (Anagnostakis et al., 2004; Dean, 2009; Glavič-Cindro and Korun, 2010; Harms and Gilligan, 2010; Lépy et al., 2010; Maringer, 2008; Maringer et al., 2008b; Pommé et al., 2009a, 2009b; Peyres and García-Toraño, 2007; Rentaría Villalobos et al., 2007; Vesterbacka et al., 2009, 2010).

In this paper, an overview on the main scientific results of the three years (2013–2016) European Metrology Research Programme's joint research project MetroNORM – Metrology for processing materials with high natural radioactivity (EURAMET, 2016) – is given.

The results and the potential impact on NORM industrial practices by implementing the developed laboratory and in-situ measurement methods, procedures, instruments, improved decay data and reference materials are discussed.

The scientific output of the joint research project has been considered in European and national standard bodies' working groups on dose assessment and classifications of emitted gamma radiation for building materials (CEN/TC 351/WG3 Radiation from construction products), technical preventive radon measures for buildings (Austrian Standards Working Group 088 14, Radon) and the determination and evaluation of the total dose due to natural radionuclides in drinking water (OENORM S5251, 2016).

2. Scientific and technical objectives of the European MetroNORM joint research project

The MetroNORM project's scientific and technical objectives are the:

1. Development of measurement systems, methods and techniques including in-situ systems which support innovative industrial processing of resources containing NORMs.
2. Design of traceable measurement procedures (as input to e.g. CEN/CENELEC standards) for industrial NORM raw materials, products, by-products, residues and waste.
3. Development and establishment of traceable metrological reference materials and standard sources needed for calibration purposes for NORM measurement.
4. Improvements to decay data for selected natural radionuclides of the ^{238}U, ^{235}U decay chains, and to the rare earth element ^{138}La (lanthanum), focusing on decay chains description and gamma-ray intensities and half-life improvement.
5. Testing of developed systems, standards and reference materials in industrial processing situations.

The JRP consortium consist of 12 National Metrology Institutes/Designated Laboratories (NMI/DI), 3 non-NMI/DI partners (REG – Research Excellent Grant holder) and 2 collaborators (IRSN, France and VUHZ a.s., Czechia).

3. Results of the joint research project

3.1. Measurement systems, methods and techniques including in-situ systems

The laboratory and in-situ measurement systems and procedures for the measurement of NORM radionuclides and the reference materials have been developed with total relative measurement uncertainties lower than 10% ($k = 1$) (e.g. Glacič-Cindro and Korun, 2014).

The project has developed new metrological methodologies, measurement instruments, standards and reference materials for measurement of natural radionuclides.

Significant improvements have been achieved by developments, designs and successful tests of a novel hand-held prototype in-situ gamma-ray measurement system, an in-situ alpha-particle spectrometry prototype by integration with a remote expert support system (Pöllänen and Siiskonen, 2014, 2015; Pöllänen et al., 2015; Pöllänen and Klemola, 2016).

Rapid detection of alpha-particle emitting radionuclides may be of utmost importance in radiation and nuclear safety, security and safeguards as well as in a nuclear emergency. In the frame of this project, a prototype in-situ alpha-spectrometry system has been developed which can be applied for all alpha emitters, measurements of flat and smooth surfaces (Fig. 1). There is no need of a vacuum pump. The system shows a reasonable energy resolution (relative energy resolution ~0.15) for radionuclide identification supported by alpha particle collimation (Fig. 2), real-time data transfer and reach back functionality. In principle, also beta particles are detectable with the system. The

Fig. 1. The MetroNORM in-situ alpha-spectrometer system Adonis in operation.

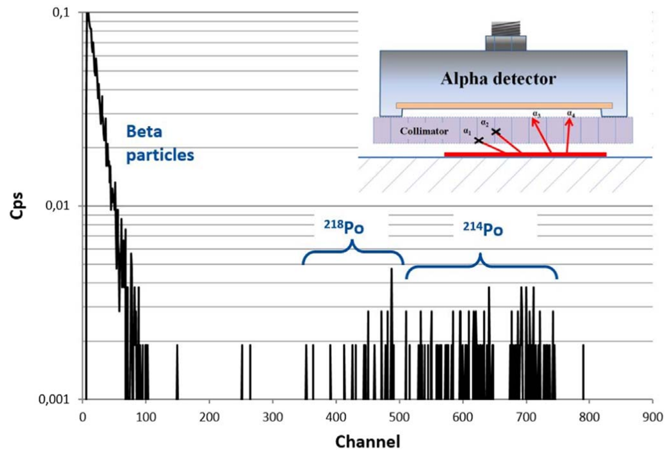

Fig. 2. Spectrum of ^{222}Rn progenies at the testing operation of the in-situ alpha spectrometer Adonis with collimator.

measurement procedure for the prototype in-situ use hand-held alpha spectrometer had been established and tested successfully.

An in-situ measurement system based on hybrid Si pixel detectors (MEDIPIX/TIMEPIX) has also been designed, developed and tested (Bulanek et al., 2015; Figs. 3 and 4). The minimum detectable total alpha and total beta sensitivities of the developed NORM pixel detector in relation to the measuring time are shown in Table 1.

An improved final NORM sample preparation stage has been developed, as well as an automated method for the final NORM sample preparation stage by extraction chromatography using UTEVA resin (Triskem International) and vacuum box, which includes an appropriate initial sample preparation stage (Katanax K2 Prime) for mixed silicaeous NORM matrices: scale, water, sand, wax, oil.

For ^{220}Rn (thoron), a production chain for the radionuclide in a vacuum chamber has been designed and set-up and the generators and ^{220}Rn samples/standards together with the chamber have been successfully tested (Sabot et al., 2015). In addition to this, a novel measurement system for the on-line determination of ^{220}Rn activity has been developed, and the results of a comparison between the ^{220}Rn standards developed has been completed (Fig. 5). The comparison showed that the relative difference is 4,7% at the maximum at $a(^{220}\text{Rn}) = 23{,}3\ \text{kBq m}^{-3}$.

3.2. *Traceable measurement procedures for industrial NORM raw material, products, by-products, residues, and waste*

37 of more than 50 registered MetroNORM stakeholders, from European NORM industries and NORM measurement laboratories have completed the project's “Questionnaire to selected industrial companies on specific methods used for characterisation residues containing natural radioactivity” or the “Questionnaire to selected laboratories contracted by industries that request analyses for characterisation of residues containing natural radioactivity”, respectively. The completed

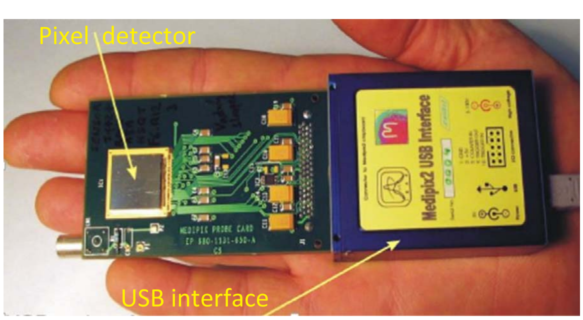

Fig. 3. Developed NORM Pixel detector with USB interface.

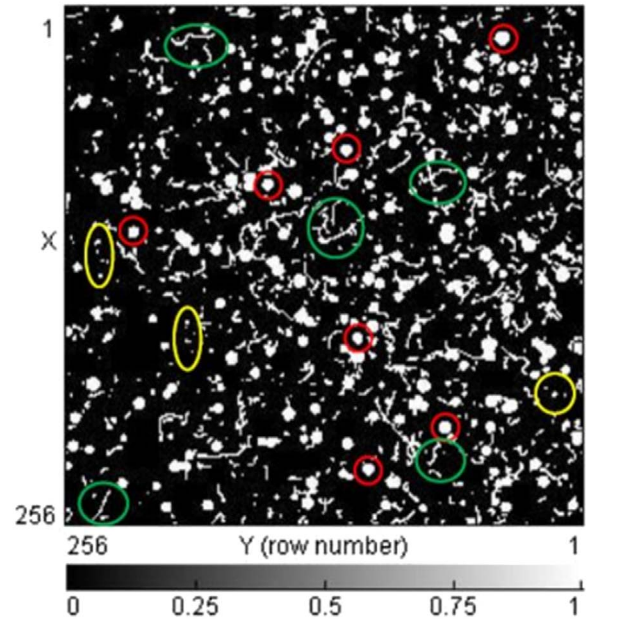

Fig. 4. NORM Alpha-particle (red, small circles), beta-particle (green, bigger circles) and gamma-ray (yellow, ovals) response at the pixel detector. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

Table 1
In-situ pixel detector system MDAs at measurement of surface activity contamination by alpha-particles and beta-particles versus measuring time.

Time of sample measurement (s)	MDA total alpha (kBq/m^{2})	MDA total beta (kBq/m^2)
1	58	84
10	6.7	14.7
100	0.96	3.5
1000	0.19	0.95

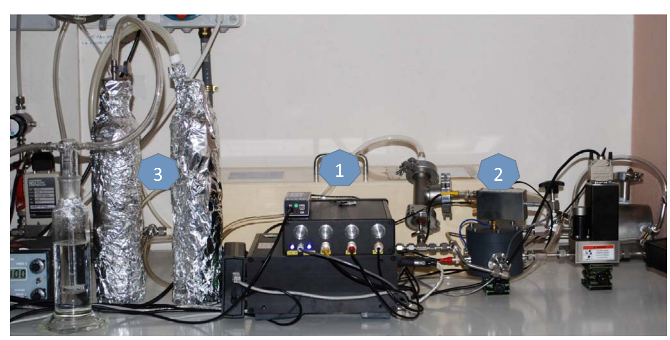

Fig. 5. ^{220}Rn measurement setup with ^{228}Th source (1), ^{220}Rn chamber (2) and gas bubblers with liquid scintillator (3).

questionnaires were then evaluated to take into account the measurement methods used by European NORM industries for residues and three main measurement methods were selected and are under evaluation for in-situ and laboratory measurements of NORM materials.

In addition, a measurement procedure for the determination of the total activity of inhomogeneously distributed NORM material in waste has been developed, as well as a protocol for testing and evaluating measurement methods used in European NORM industries for waste (Fig. 6; Glavič-Cindro and Korun, 2014).

Novel methods for the measurement of airborne radionuclides using the prototype pixel detector (MEDIPIX/TIMEPIX), and for use of the prototype in-situ use hand-held alpha spectrometer (both from objective 1) have been successfully created. A report on best practice

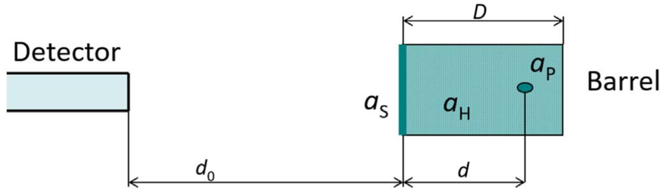

Fig. 6. Detection of inhomogeneity in NORM waste drums: Three distinct contributions to radioactivity distribution supposed: surface (a_S), homogeneous (a_H) and point source (a_P) contributions.

methods for radioactivity measurements for laboratory analysis has also been prepared.

Further to this, measurement procedures for the in-situ analysis of radon in waterworks (Fig. 7) and for in-situ analysis of radon emanation from building materials (Fig. 8) have been established.

3.3. Traceable NORM reference materials and standard sources

New candidate reference materials have been prepared for the traceable calibration of NORM measurements using the project partners' national standards. So far, the project partners have determined the volumes, natural radionuclides activity concentrations, matrices and the composition of the candidate reference materials. The candidate reference materials include; Ta/Nb (tantalum/niobium) ore processing, residue/waste from titanium dioxide (TiO_2) production, coal ash and tuff used in building products, residue/waste from phosphorgypsum processing, building aggregates, ion exchange resins from the water industry, and iron oxide/manganese dioxide ($FeO(OH)/MnO_2$) sludge from the water industry and oil waste.

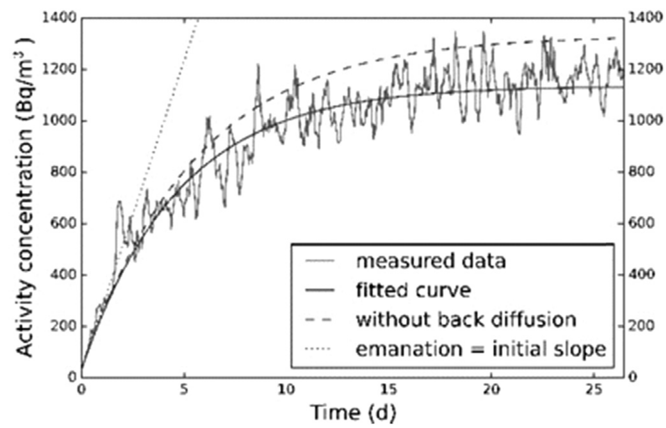

Fig. 8. Radon emanation measurement of building materials.

NORM standard reference materials and sources for calibration of laboratory instruments and in-situ measurement instruments - covering the NORM radionuclides ^{238}U, ^{235}U, ^{226}Ra, ^{210}Pb, ^{228}Ra, ^{228}Th, ^{208}Tl, ^{228}Ac, ^{214}Bi, ^{214}Pb, and ^{40}K - have been prepared and applied successfully.

In total 10 calibration, standard sources and reference materials for gamma-ray spectrometry and alpha spectrometry measurement and for in-situ measurement systems for NORM have been developed by the project. The reference materials and standard sources have been characterised completely. The activity concentrations of natural decay chain radionuclides range from ~0.1 Bq/kg to ~100 kBq/kg. Further to this, two inter-laboratory comparisons using the standard sources and reference materials have been successfully carried out. Additionally,

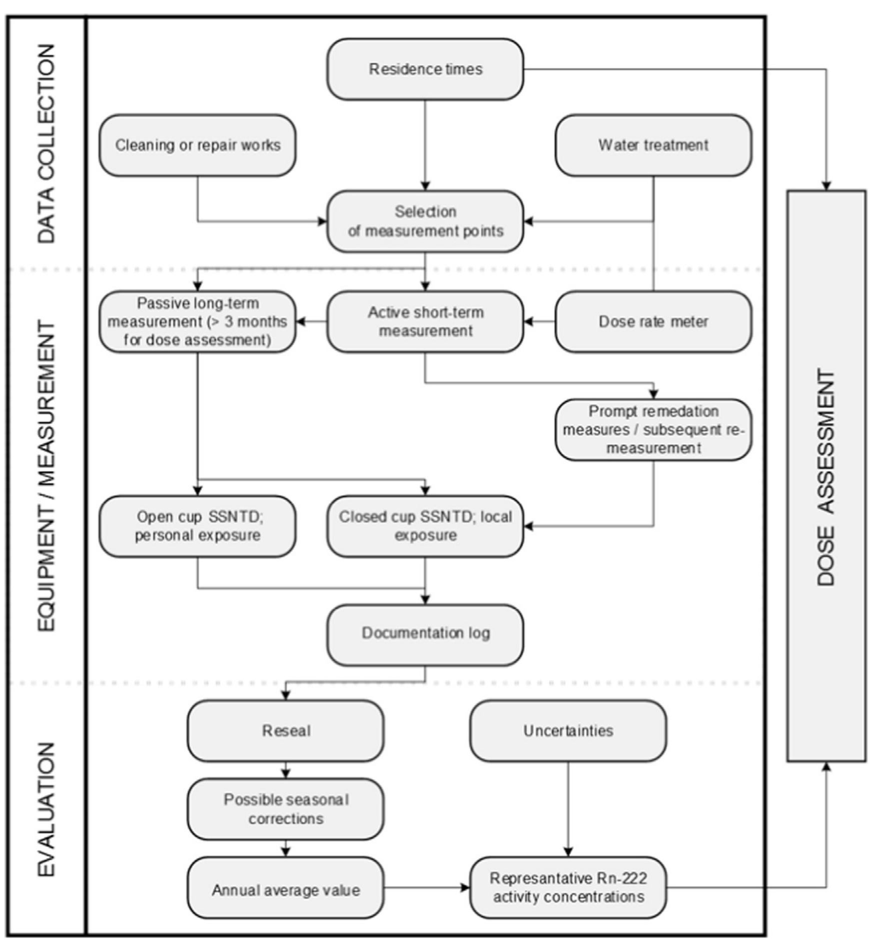

Fig. 7. Scheme of the in-situ method for exposure and dose assessment of radon in drinking water plants.

two standard sources for the calibration of the pixel detector have been completed and measured.

3.4. Improved decay data for selected natural radionuclides

The revision of nuclear data (Bé et al., 2011) for selected natural radionuclides is being done so that as many descendants of uranium and thorium decay chains can be accurately measured as possible. To improve the required NORM related decay data, special radionuclide sources with the radionuclides ^{235}U, ^{227}Ac, ^{226}Ra and ^{210}Pb and have been carefully prepared and improved decay data of the selected NORM radionuclides' has been prepared (Rodrigues et al., 2016). The calculation of the improved beta spectrum of ^{138}La has been finished and the gamma-ray spectra of 4 selected key NORMs have been measured and evaluated. A revised decay scheme for ^{138}La including updated decay data has been established. The improved calculation of the beta energy spectrum of ^{138}La based on a 2nd forbidden unique transition shows better agreement to the experimental data both in the low-energy region 0–50 keV and at intermediated energies 100–200 keV. In addition, the complex gamma-ray spectra of selected NORM key-materials have been measured and evaluated.

3.5. Testing of developed systems, standards and reference materials in industrial processing situations

In the final project phase, site specific verification criteria, requirements and procedures for selected end-user sites have been developed, for testing the in situ systems, methods and reference materials from objectives 1, 2 and 3. The location, instrumentation and procedures for these on-site/in situ tests have been prepared carefully. These final on-site tests have been successfully carried out:

- Development of in-situ alpha spectrometer (Roy Pöllänen et al., STUK - Radiation and Nuclear Safety Authority, Finland)
- Pixel detectors and their application in NORM Industry (Jiří Hůlka et al., SURO - National Radiation Protection Institute, Czechia)
- ^{210}Pb activity determination using gamma-ray spectrometry (Mikael Hult et al., JRC - Joint Research Centre, Belgium)
- Direct method for ^{210}Pb activity concentration determination (Boguslaw Michalik et al., GIG - Central Mining Institute, Poland)
- On-site verification of a measurement procedure for radon in waterworks (Michael Stietka et al., BOKU - University of Natural Resources and Life Sciences, Austria)
- Method for determination of the total activity of radioactive waste in drums (Branko Vodenik et al., IJS - Jozef Stefan Institute, Slovenia)

4. Impact of the three years research work

Further to these activities, the project has established an Advisory Board of experts from the IAEA, International Committee for Radionuclide Metrology (ICRM), European Cooperation in Science and Technology (COST), European ALARA Network for NORM (EAN-NORM), and NORM industries who are helping to steer the project towards meeting end-users needs.

The results of the project have also been disseminated to European and national standardisation bodies' and working groups such as CEN/TC 351 Construction products – Assessment of release of dangerous substances WG3 on radiation form construction products, CEN/TC 45 Nuclear Instrumentation, the ASI – Austrian Standards Institute, and the EC Group of Experts established under Article 31 of the Euratom Treaty.

The metrological results of the JRP have been considered in the European COST Network NORM4BUILDINGS (European Cooperation in Science and Technology, Action TU1301). This COST initiative stimulates the collaboration of scientists and stakeholders (industries and regulators) in gathering knowledge, experiences and technologies, to stimulate research on the reuse of residues containing enhanced concentrations of natural radionuclides (NORM) in tailor-made building materials in the construction sector while considering the impact on both external gamma exposure of building occupants and indoor air quality. The results of MetroNORM, especially the newly developed NORM in-situ measurement methods and traceable reference materials and sources will help to improve the radiological impact assessment models for the reuse of NORM residues in building materials.

The first project workshop was successfully held as part of the EU-NORM 2 conference in Prague, 19 June 2014. In this workshop project partners, together with more than 40 stakeholders and end-user presented and discussed relevant issues for NORM facilities, nuclear regulators, standardisation bodies and measurement device and calibration source manufacturers. The second workshop for stakeholders and industrial end-users took place in Lisbon on 11–12 March 2015 and the final project stakeholders' workshop had been held on 22 June 2016 at the famous Hotel Metropole in Brussels.

Furthermore, the results of the project have been presented to metrologists, stakeholders, regulators and end-users at international conferences, such as the 20th International Conference on Radionuclide Metrology ICRM 2015 and the International Conference on Low-Level Radionuclide Metrology Techniques (ICRM-LLRMT 2016). A joint workshop together with the EU Cost network NORM4BUILDINGS was held with more than 50 participants of European NORM industry companies and radiation protection regulators.

To address the education of end-users, regulators and the public, an open access NPL e-learning course on NORM metrology and measurement has been established by the JRP partners (http://www.npl.co.uk/commercial-services/products-and-services/training/e-learning/naturally-occurring-radioactive-materials/).

A concept and training material for a post-graduation university NORM course has also been developed. These materials include:

- The hazards and risks associated with work involving NORM raw materials, by-products, residues and wastes
- National and international requirements and recommendations
- Best practices and state-of-art measurement systems regarding NORM monitoring at industry
- Measurement systems, standard sources and reference materials to perform traceable NORM analysis.

The NPL e-learning course, the established university NORM course training materials for end users and NORM industry stakeholders and the published scientific papers of the JRP results create sustainably impact of the JRP in the next years beyond the end of the JRP.

5. Conclusions

The developments of the EMRP IND 57 MetroNORM European Joint Research Project have been presented at 40 scientific conferences and published by 18 peer-reviewed scientific journal papers (http://www.metronorm-emrp.eu: Documents section). The scientific, technical and operational findings of the MetroNORM JRP give essential improvements for traceable instrument calibrations and activity measurements of natural radionuclides in the European NORM industry.

The outcome of the JRP provides a metrological sound basis for radiation protection of European citizens in the NORM industry field. This is the objective of the new EURATOM Basic Safety Standards Directive (EC, 2013), which has to be implemented by national legislatives in the coming years.

Reliable, traceable and consistent NORM activity measurements are necessary in order to optimize counter measures for the reduction of the exposure to the public and to NORM industry workers. This will then reduce the related follow-up costs. The lower the uncertainties of the NORM activity measurement results, the lower is the risk of possible over-responses of counter measures, with low radiological but high economic impact.

Traceability of all NORM calibrations and measurements and dose assessment steps is necessary, in particular given their possibly important economic and political impact, yet methodically different. Therefore, an overall conceptual outcome of this project is to support the establishment of an expanded metrological NORM framework, which helps the European NORM industry to remain globally competitive.

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