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Mass spectrometry developments of ^{232}Th and ^{238}U radiopurity measurements for LEGEND

Jakub Kaizer[1] · Marek Bujdoš[2] · Raffaele Buompane[3] · Silvia Dulanská[4,5] · Miroslav Ješkovský[1] · Ivan Kontul'[1] · Stefano Nisi[6] · Pavel P. Povinec[1] · Carmina Sirignano[3] · Filippo Terrasi[3] · Jakub Zeman[1]

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Abstract
The LEGEND collaboration has been developing a ^{76}Ge-based double-beta decay experimental program where precise radiopurity measurements of ultraclean materials are crucial. Ultralow concentrations of thorium and uranium, the main contributors to the detector background via their decay products, can be determined by inductively coupled plasma mass spectrometry (ICPMS) and accelerator mass spectrometry (AMS). Here we shall present recent developments in thorium and uranium mass spectrometry methods, together with basics of separation chemistry applied to process different samples. The new possibilities to measure ^{232}Th and ^{238}U by ICPMS and AMS at the Comenius University in Bratislava are discussed as well.

Keywords AMS · ICPMS · Thorium · Uranium · LEGEND · Radiopurity

Introduction

In recent years, several large-scale physics experiments have been designed or put in operation to search for neutrinoless double beta decay, which, if observed, would enable scientists to answer the question if neutrino is a so-called Majorana particle and what its mass is [1–6]. One of such efforts is the world LEGEND (Large Enriched Germanium Experiment for Neutrinoless Double Beta Decay) experiment which uses high-purity germanium detectors made of material with the enriched level of ^{76}Ge [7, 8]. The first phase, LEGEND-200, is a successor of the GERDA experiment [9], operating with its infrastructure at the Gran Sasso National Laboratory (LNGS) in Italy and also implementing the knowledge obtained from the development of another germanium double beta decay detector, MAJORANA DEMONSTRATOR [10]. The LEGEND-200 will comprise detectors containing roughly 200 kg of enriched Ge, reaching the sensitivity for neutrinoless double beta decay at the level of $> 10^{27}$ yr after 5 years of data taking, while having the overall background below 2.5×10^{-4} counts keV^{-1} kg^{-1} yr^{-1} [11]. Next, the LEGEND collaboration plans to develop and construct an experiment that will use detectors with around 1000 kg of germanium with the total background of less than 1.0×10^{-5} counts keV^{-1} kg^{-1} yr^{-1} at the same time. LEGEND-1000 is expected to run for at least ten years, resulting in a limit for the half-life of neutrinoless double beta decay for ^{76}Ge beyond 10^{28} yr [12].

The background of complex and sensitive detector consists of various components from different sources, naturally occurring thorium ^{232}Th ($T_{1/2} = 1.40 \times 10^{10}$ yr) and uranium ^{238}U ($T_{1/2} = 4.47 \times 10^{9}$ yr) and their short-lived progenies being one of the most important contributors. In the case of LEGEND, radiation from daughter radioisotopes of thorium and uranium will make up to ~ 20–30% of the overall

✉ Jakub Kaizer
jakub.kaizer@uniba.sk

1 Center for Nuclear and Accelerator Technologies (CENTA), Faculty of Mathematics, Physics and Informatics, Comenius University, 84248 Bratislava, Slovakia

2 Faculty of Natural Sciences, Comenius University, 84215 Bratislava, Slovakia

3 Department of Mathematics and Physics, University of Campania "Luigi Vanvitelli", 81100 Caserta, Italy

4 Institute of Medical Physics, Biophysics, Informatics and Telemedicine, Faculty of Medicine, Comenius University, 81372 Bratislava, Slovakia

5 Faculty of Public Health, Slovak Medical University, 83303 Bratislava, Slovakia

6 Laboratori Nazionali del Gran Sasso, Istituto Nazionale Di Fisica Nucleare, 67010 Assergi, AQ, Italy

background, depending on the phase of the experiment [12]. Natural thorium and uranium are part of each material used for construction of the experiment, thus the information about their content is crucial for correct estimation of the background. Some materials, which are situated far from the sensitive volume of detector (e.g., cables, optical fibers), can be screened by non-destructive gamma spectrometry. More critical parts, such as germanium crystals and detector mounts, usually require the application of mass spectrometry techniques, which are generally much more sensitive for long-lived radionuclides than conventional radiometric methods [13–15]. Inductively coupled plasma mass spectrometry (ICPMS) has become a routine method for radiopurity measurements of high-purity materials, offering relatively quick and inexpensive analysis down to 10^{-15} g g^{-1} [16–18]. An even greater potential has accelerator mass spectrometry (AMS), whose exceptional limit of detection is given by the removal of interfering ions due to accelerator-based stripping and multiple mass separation steps. However, if one does not deal with specific materials [19], sample preparation is obligatory for both ICPMS and AMS.

The scientific program of the Comenius University in Bratislava has been devoted to – among others – nuclear physics and environmental studies that have been based on the determination of low-level activities of radionuclides and concentrations of elements (stable isotopes). At the Faculty of Mathematics, Physics and Informatics, two shielded HPGe detectors are in operation, which have been used for low-background gamma spectrometry measurements of natural and anthropogenic radionuclides in samples of various origin and rare events search [20–24]. Centre for Nuclear and Accelerator Technologies (CENTA) was established at the university ten years ago, comprising a tandem accelerator laboratory that has been used intensively for ion beam and mass spectrometry analyses [25–28], including AMS measurements of ^{10}Be [29] and testing of uranium target matrices [30]. On top of that, the application of ICPMS for elemental composition determinations has been frequently performed at the Faculty of Natural Sciences of the Comenius University [31, 32].

LEGEND-200 shall start acquiring data soon, which means that all important parts of the detectors have been analyzed for the content of critical radionuclides and have met the required limits. However, one can reasonably anticipate that some of the parts may need replacement or upgrade as the experiment proceeds with time. Moreover, the LEGEND-1000 phase is only in its early development stage; many of the materials selected for the commissioning of the experiment will first have to be screened. Thus, the radiopurity assessment program of LEGEND is far from being over and should be intensified with the increasing demand for (ultra)clean materials. Here we present recent developments in thorium and uranium determinations by ICPMS and AMS, together with basics of separation chemistry applied to process different sample matrices. The new possibility to measure ^{232}Th and ^{238}U by ICPMS and the development of AMS at the Comenius University in Bratislava is also discussed.

Developments in sample preparation for natural Th and U mass spectrometry analysis

In collaboration with LNGS [33, 34], we have developed a simple extraction chromatography method based on the simultaneous separation of Th and U from metallic matrices with the use of TRU Resin (TrisKem International) that can now be utilized in the CENTA sample preparation laboratory. In short, sample is rinsed/etched to remove surface contamination and dissolved in ultrapure concentrated inorganic acid (HNO_3/HCl). Diluted sample is loaded on a pre-treated TRU Resin chromatographic column which is then washed to separate matrix. Consequently, thorium and uranium are eluted by ammonium oxalate solution, which is then analyzed directly by ICPMS. Another option is to further process solution to obtain a target for AMS which we indeed exploited in our next development phase (see below). The method described above has been proven to reliably extract thorium and uranium with a yield of $\geq$ 90%, regardless the type of sample and its processed amount. Various types of solid materials (powders, scobs, wires, and pellets) have been treated to efficiently recover Th and U from metallic materials such as copper or aluminum; with slight modifications, the method could be usable for an even wider spectrum of metals/alloys, e.g., iron or steel, which are often exploited in experiments probing for rare nuclear processes. Procedural blanks on the level of $\leq$ 0.8 pg g^{-1} for ^{232}Th and ^{238}U can be achieved if one takes suitable countermeasures to prevent contamination of samples during their treatment (e.g., working in a clean environment, using only ultrapure chemicals, water and whole-body protection, extensive rinsing of glassware/labware, control of extraction chromatography resins). Specific precautions are needed for AMS; target holders (cathodes) and targets wheel, which are sputtered together with samples during the ion production, should be made of high-purity material. Very good quality was confirmed in the case of copper AMS target holders manufactured by National Electrostatic Caorporation (NEC, Middleton, USA), showing mean ^{232}Th and ^{238}U concentrations ~ 2 pg g^{-1}.

Polymeric (plastic) materials are quite common parts of large-scale experiments such as LEGEND but generally require a different approach in treatment for radiopurity measurements than metallic components. We have tested

a procedure for processing various polymeric foils (three Mylar and two polyethylene samples), with a main goal of determining the memory effect of containers used for combustion and its contribution to overall ^{232}Th and ^{238}U blanks. Samples were combusted in fused quartz crucibles (Thermo Fisher Scientific) in a furnace at 650 °C for 6 h; residual material was dissolved by the addition of 10 mL of 10% HNO_3 to the crucible, which was then heated to 80 °C for 1 h. Consequently, two volumes of 10 mL 10% HNO_3 were heated under the same conditions in the empty crucibles prior to each combustion and after the last one to obtain blank solutions that were later analyzed by ICPMS at LNGS, together with sample solutions. The results showed that the memory effect of the crucibles is more or less negligible and that they can contribute to contamination by thorium and uranium with an average value of 1.2–2.7 pg g^{-1} (Fig. 1) if we consider only the first two rinses as they were not affected by the subsequent combustions. Interestingly, the fused quartz crucibles seemed to be a greater source of ^{232}Th than ^{238}U.

Developments of AMS for natural Th and U determination

The determination of ultralow concentrations of ^{232}Th and ^{238}U by AMS is one of the very important topics in radiopurity assessment due to the prevailing demand for high-purity materials that can only be effectively screened by mass spectrometry techniques. Therefore, we have taken the first steps in the development of this method in collaboration with the CIRCE laboratory, which has a long history of AMS measurements of heavy isotopes [35, 36]. For testing purposes, we prepared a series of targets from solutions from the previous development step that contained known concentrations of natural thorium and uranium (verified by ICPMS at LNGS) and that were spiked with the ^{229}Th tracer. The preparation was based on co-precipitation of radionuclides of interest on the iron oxide matrix. This was achieved by addition of Fe^{3+} carrier, purified from ^{232}Th and ^{238}U using UTEVA Resin (TrisKem International), to the solutions and raising their pH to 8–10 with concentrated NH_4OH. The obtained precipitates were centrifuged, dried, baked at 550 °C and pressed in copper target holders (from NEC) which were then analyzed by AMS.

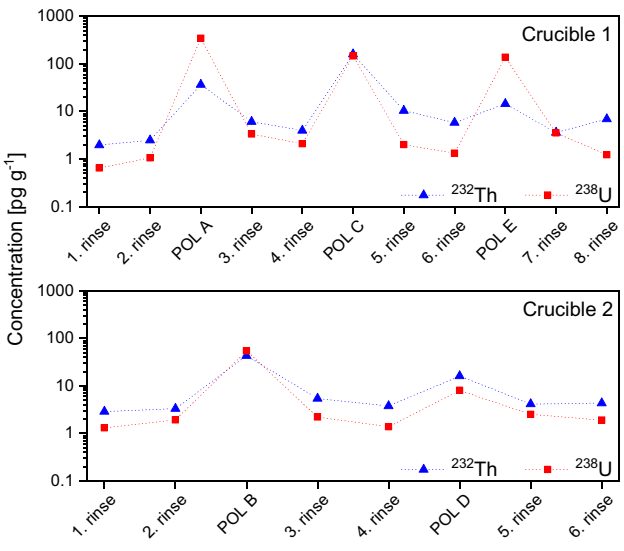

Fig. 1 Concentration of ^{232}Th and.^{238}U in 10% HNO_3 rinses from two fused quartz crucibles before and after combustion of various polymeric (plastic) samples (POL A–E)

Measurements of thorium and uranium targets were conducted with the 3 MV tandem accelerator system at CIRCE. The tuning of the AMS system was performed using ^{238}U from standard cathodes containing pure uranium oxide of a natural origin, however, we decided to detect ^{235}U ions from samples instead as their ^{238}U signal outreached the count-rate limit of the ionization chamber; the ^{238}U levels were calculated according to the natural isotopic ratio $^{235}U/^{238}U \approx 7.26 \times 10^{-3}$. Due to the unavailability of a suitable uranium tracer (e.g., ^{233}U), we determined the isotopic ratios $^{229}Th/^{232}Th$ and $^{229}Th/^{235}U$ while applying corrections for different production rates of thorium and uranium ions in the source. For injection, molecular oxides $Th^{16}O^-$ and $U^{16}O^-$ were selected which were then stripped to single atoms with a charge state of 5 + and accelerated to the energies 16.70, 16.50 and 16.28 MeV for $^{229}Th^{5+}$, $^{232}Th^{5+}$ and $^{235}U^{5+}$ ions, respectively (Fig. 2).

A comparison of the results from the AMS and ICPMS measurements of ^{232}Th and ^{238}U in the testing samples is shown in Fig. 3. The values represent the total amounts of ^{232}Th and ^{238}U in the AMS targets and in the solutions analyzed by ICPMS, discussed in the previous chapter. Unlike for ^{232}Th, where its chemical yield was controlled by ^{229}Th, the transfer of ^{238}U from solutions to targets was only anticipated to be efficient. However, a reasonably good correlation between the methods (r = 0.9967 if obvious outliers are excluded) suggests that the uranium yield was indeed very high. Except for one case, the outliers represent samples whose analysis might have been affected by their contamination during the pressing, or by crosstalk and memory effect of the target wheel; the issue was addressed in the case of later samples. The relative statistical uncertainties of the AMS determinations were in the range of 7.2–10.3% for ^{232}Th and 6.8–19.1% for ^{238}U; slightly higher values in the case of uranium were probably caused by the absence of a tracer. By measuring one of the procedural blank solutions, we determined the limit of detection to be 0.4 pg of actinide per target. From the total detected counts for this sample, we

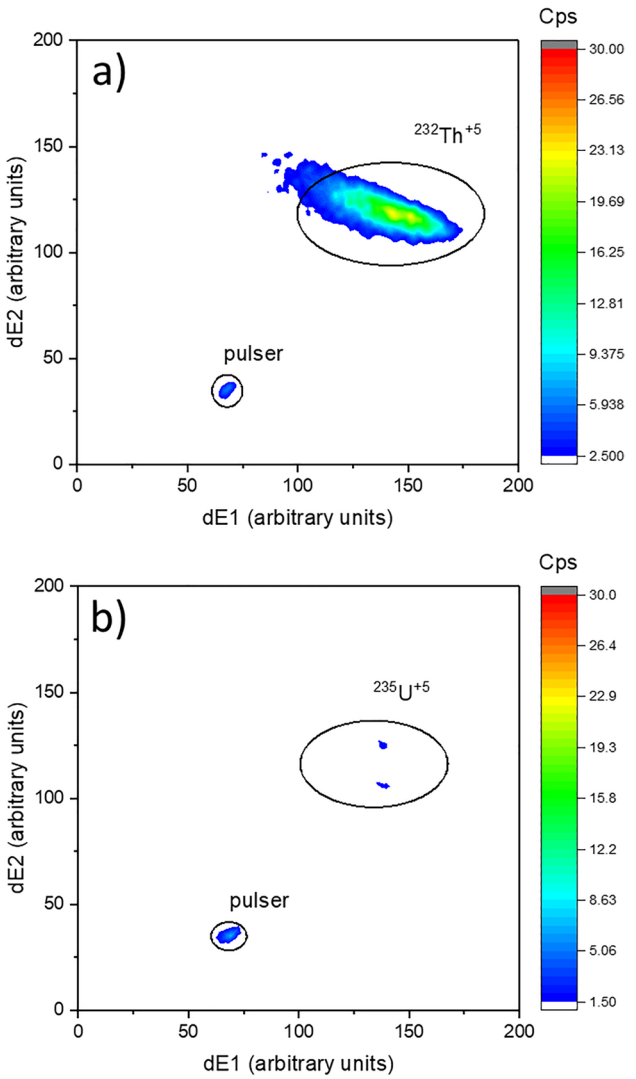

Fig. 2 Spectra obtained from measurements of natural thorium (**a**) and uranium (**b**) ions in one of the testing samples, analyzed by the CIRCE AMS system

estimate the instrumental limit of detection to be one order of magnitude lower.

The aforementioned experiments have the potential to serve as a solid basis for further development of AMS analysis of natural thorium and uranium. In August 2022, the CENTA tandem laboratory was upgraded with a new AMS line supplied by NEC, which comprises a 90° analyzing electromagnet with a high resolution ($ME/Z^2 = 176$ MeV-amu), two 45° electrostatic analyzers, and a 4-anode ionization chamber for detection of ions of interest, including actinides (Fig. 4). The entrance window of the ionization chamber can be mounted by foils of different materials to suppress isobaric interferences. Another important part of the installation was a fast-bouncing system that enables switching between injected ions in a matter of microseconds, which is essential for precise measurements of isotopic ratios. Moreover, the accelerator has been equipped with an electrostatic quadrupole that significantly improved the possibility to focus the ion beam after its acceleration. After the necessary setting and optimization of the upgraded tandem system, we shall follow up on the already obtained results and finalize the development of the AMS method for ultra-low determination of ^{232}Th and ^{238}U for material screening in the near future.

ICPMS capabilities for natural Th and U determination

Several companies have been developing and commercializing stand-alone ICPMS instruments for elemental and isotopic analysis which, in comparison to AMS, have had a lower complexity and a simpler operation. At the Faculty of Natural Sciences, there are two ICPMS machines in operation that can be utilized for the determination of thorium and uranium. The first one is a quadrupole mass spectrometer PerkinElmer Elan 6000 with a simultaneous extended dynamic range detector (dual-stage discrete dynode electron multiplier). The instrument is capable of effectively measuring almost all isotopes (up to 270 amu), and it is equipped with a HF-resistant Scott-type spray chamber, which can be especially useful when dealing with samples that require the application of highly concentrated hydrofluoric acid for their dissolution. (e.g., pure germanium oxide intended for the production of detectors). Even though these types of spectrometers have only one mass analyzer and do not contain any reaction/collision cell for suppression of isobaric interferences, their limit of detection for ^{232}Th and ^{238}U determinations (~ 1 pg g^{-1}) is generally sufficient for radiopurity measurements of non-critical components.

In 2016, the university acquired a new ICPMS machine Thermo Scientific iCap Q which works with a single quadrupole, a He collision cell, and a dual-mode discrete dynode electron multiplier for ion detection. The spectrometer has been shown to provide precise and accurate results for natural thorium and uranium at μg L^{-1}/μg kg^{-1} levels, however, the declared limits are by a few orders of magnitude lower. To test them, we prepared and analyzed a series of calibration solutions containing 0–250 pg mL^{-1} of thorium and uranium. As expected, we obtained a sensitivity of ~ 1700 and 3300 cps per 10 pg mL^{-1} of ^{232}Th and ^{238}U in the standard mode (STD), respectively, while these values were found to be approximately 30% higher when the helium collision cell was used. According to the calibration curves shown in Fig. 5, we were able to achieve the instrumental limit of detection of 0.39 pg mL^{-1} for ^{232}Th and 0.023 pg mL^{-1} for ^{238}U in the standard mode.

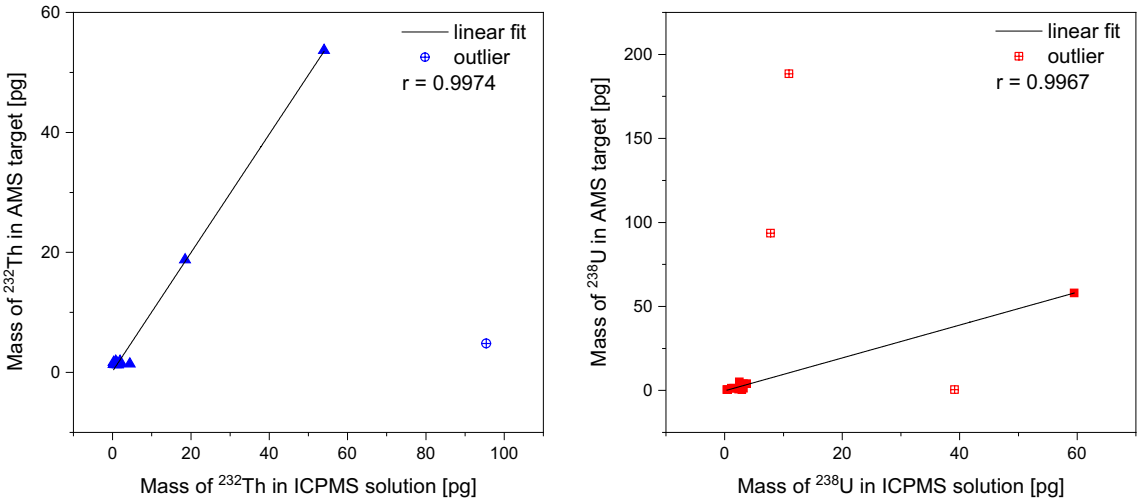

Fig. 3 Comparison of the ICPMS and AMS results for the determination of natural thorium (left) and uranium (right). The linear fit lines were constructed excluding obvious outliers

Fig. 4 Newly installed AMS parts of the tandem accelerator system at the CENTA laboratory: 4-anode ionization chamber (top left), high-resolution analyzing magnet (top right), and two electrostatic analyzers (bottom)

Thorium concentrations were determined with rather high standard deviations; however, this can be addressed by setting a longer time for stabilization of the detected signal.

Outlook

LEGEND represents the state-of-art experiment that has undoubtedly a potential to prove the existence of neutrinoless double beta decay. The success of the collaboration depends on many things, one of them being the ability to design and construct a detector that operates with virtually no background radiation. This strict demand has been fulfilled in the case of LEGEND-200, which should provide first results in the upcoming years. On the other hand, the task will be more challenging for LEGEND-1000 which is planned to work with five times more mass of germanium while at the same time having the background lower by one order of magnitude. After the start of its main part, the screening campaign for LEGEND-1000 will take some time before all appropriate materials will be identified, even if it is expected that at least twenty gamma spectrometry, neutron activation analysis and mass spectrometry facilities could participate in radiopurity measurements. Due to their ubiquity and relatively high abundance, naturally occurring thorium and uranium will be one of the most important radionuclides to target.

Comenius University in Bratislava has recently invested in the development of mass spectrometry techniques which have become mandatory for ^{232}Th and ^{238}U analyses in the case of large physical experiments searching for rare nuclear processes. Because these methods require samples to be treated and transformed into solutions or targets, we have tested procedures for the decomposition of metallic and polymeric materials and the consequent separation of thorium and uranium. Regarding the analytical infrastructure, two ICPMS instruments are now available to screen samples with actinide concentrations

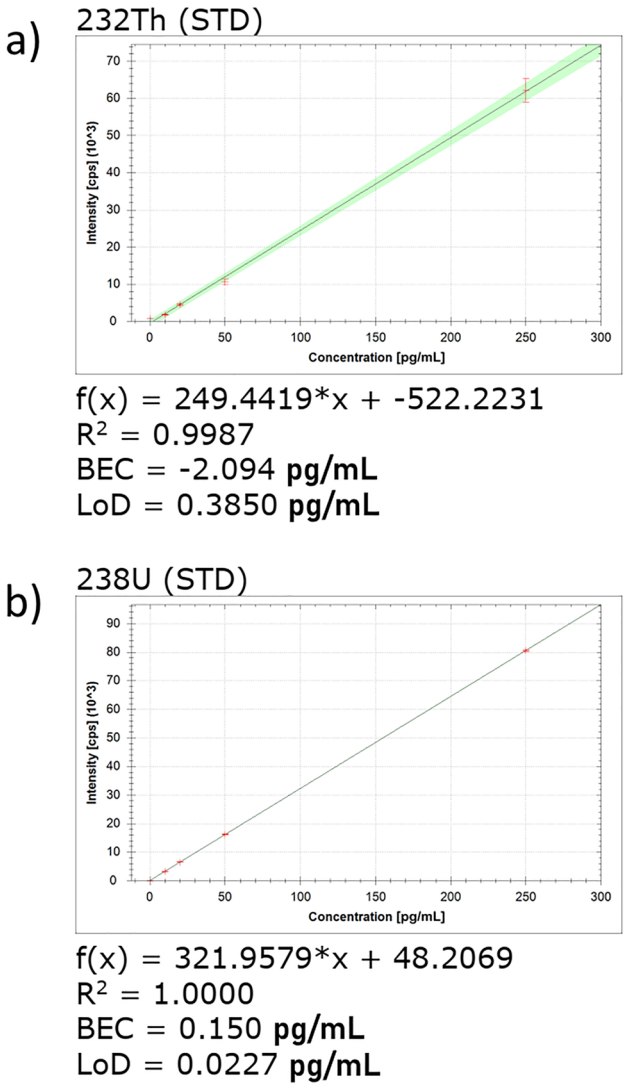

Fig. 5 Calibration curves for ^{232}Th (**a**) and ^{238}U (**b**) concentrations in solutions analyzed by Thermo Scientific iCap Q

of > 1 pg g^{-1} or even less, depending on the quality of simultaneously prepared blanks. It is assumed that this limit could be further enhanced by utilizing the AMS line that has been recently installed at the CENTA tandem laboratory, bringing to the material screening program of the LEGEND collaboration another possibility for ultra-trace analysis of natural thorium and uranium. It is planned that the CENTA AMS laboratory will be the first world laboratory devoted to the radiopurity research.

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Data availability No additional data sets were generated or analyzed during the current study.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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