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Radiopurity assay of archaeological lead and related compounds for astroparticle physics by mass spectrometry and gamma-ray spectrometry

Luigi Cappelli [a], Massimiliano Clemenza [b], Federico Filippini [c], Matthias Laubenstein [a], Stefano Nisi [a,*], Lorenzo Pagnanini [d], Luca Pattavina [c]

[a] INFN - Laboratori Nazionali del Gran Sasso, 67100, Assergi, L'Aquila, Italy
[b] INFN - Sezione Milano-Bicocca, 20126, Milano, Italy
[c] Università di Milano-Bicocca, Dipartimento di Fisica, 20126, Milano, Italy
[d] Gran Sasso Science Institute, 67100, L'Aquila, Italy

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ABSTRACT

A novel and rapid method is introduced for determining thorium (Th) and uranium (U) concentrations in archaeological lead and related compounds to be used in astroparticle physic. This method involves chromatographic pre-concentration and inductively coupled plasma mass spectrometry (ICP-MS). Combined data from mass spectrometry and gamma-ray spectrometry measurements are presented. Excellent results were achieved in terms of reliability and sensitivity ($ng \cdot kg^{-1}$), even without introducing Th and U isotopes as tracers.

1. Introduction

The detection of low-energy neutrinos and dark matter (DM) particles is among the most pressing challenges in astroparticle physics [Billard, 2022]. These searches require the development of ultra-sensitive detectors, with possibly sub-keV energy threshold, operating in extremely low-background environments. The RES-NOVA project [Pattavina, 2020; Pattavina et al., 2021] proposes a novel cryogenic detector concept based on $PbWO_4$ crystals produced from archaeological lead (Pb), operated at temperatures that are few mK above the absolute zero [Beeman et al., 2022]. The project aims to detect neutrinos via Coherent Elastic Neutrino-Nucleus Scattering (CEνNS) and probe dark matter interactions through nuclear recoils [Akimov et al., 2017].

A critical innovation of RES-NOVA lies in the use of archaeological Pb, a material that, after separation of the parent nuclides Thorium and Uranium, has been naturally shielded from cosmic radiation for centuries (underwater in shipwrecks) such that the residual radioactivity has almost completely decayed over two millennia. This long-term storage results in a significant reduction in naturally occurring isotopes, especially ^{210}Pb, making it ideal for low background applications (Pattavina, 2019). Compared to commercially available low-radioactivity Pb, archaeological Pb can exhibit radioactivity levels up to four orders of magnitude lower, thereby minimizing one of the dominant background contributions in rare-event searches.

This work presents the radiopurity assay of archaeological Pb, tungsten oxides, lead tungstate powders, and crystals using ICP-MS and gamma-ray spectrometry. A novel pre-concentration method using TRU chromatographic resins for U and Th, provided by Triskem (Triskem International, France), is developed and validated to improve detection sensitivity. Results are discussed to compare both analytical techniques and support the selection of materials for the RES-NOVA experiment.

2. Material and methods

2.1. Analytical strategy and sample preparation

The aim of this work is to develop analytical methods capable of

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* Corresponding author.
E-mail addresses: luigi.cappelli@lngs.infn.it (L. Cappelli), massimiliano.clemenza@mib.infn.it (M. Clemenza), f.filippini2@campus.unimib.it (F. Filippini), matthias.laubenstein@lngs.infn.it (M. Laubenstein), stefano.nisi@lngs.infn.it (S. Nisi), lorenzo.pagnanini@lngs.infn.it (L. Pagnanini), luca.pattavina@unimib.it (L. Pattavina).

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measuring the radiopurity of $PbWO_4$ crystals and raw materials such as archaeological lead and tungsten oxide. Since these compounds have different chemical characteristics, the sample treatment procedure must be tailored for each of them. To achieve the best analytical sensitivity, the common approach for all samples is the need to pre-concentrate thorium and uranium using chromatographic extraction resins to separate them from the matrix. Cartridges containing TRU resins were selected for this purpose due to their strong affinity for Th and U and their high selectivity (Horwitz, 1993). Deionized water (18.2 MΩ cm) produced by a Milli-Q® Element system and ultrapure nitric acid purified using a sub-boiling distillation system were used in all chemical procedures. Sample treatment and measurements were conducted in an ISO6 cleanroom at LNGS to avoid contamination and ensure trace-level detection limits.

2.2. Instrumentation and isobaric interference resolution

A double-focusing High-Resolution Inductively Coupled-Plasma Mass Spectrometer (HR-ICP-MS), model Element2 (Thermo Fisher Scientific, USA), was used to analyze all blanks, reagents, and samples. The instrument was daily tuned to ensure maximum sensitivity and signal stability. Signals of Th and U were acquired in both Low Resolution (LR) and Medium Resolution (MR) modes to monitor potential polyatomic isobaric interferences caused by residual matrix components.

Indeed, the polyatomic species $^{184}W^{16}O_3^+$, which can be formed in the plasma torch, overlaps with the ^{232}Th signal in LR mode. However, MR mode with a resolution power of 4000 (RP = m/Δm) allows for effective separation of this interference that needs a value for RP of at least 2266. This capability is critical when analyzing samples that contain tungsten, such as WO_3 and $PbWO_4$.

2.3. Sample treatment procedures

2.3.1. Lead samples

Few grams of metallic or powdered lead are easily soluble in a 4 M ultrapure nitric acid solution up to a concentration of 20 % by weight, avoiding the precipitation of $Pb(NO_3)_2$. In case of crystallization, the precipitate can be dissolved by adding a small volume of ultrapure water. The solution obtained is then loaded onto the TRU® resin cartridge to extract Th and U before measurement with ICP-MS.

2.3.2. Tungsten oxide and lead tungstate samples

WO_3 and $PbWO_4$ are difficult to dissolve in acidic solutions due to the precipitation of tungstic acid (H_2WO_4), which may co-precipitate Th and U. This behavior is attributed to similar chemical properties between Th, U, and W, and their ability to intercalate in the layer lattice of tungstic acid [Wells,1986]. Moreover, the presence of many oxygen atoms with free electron pairs in the lattice could promote the forming complexes following mechanism similar what occurs for organic compounds rich of oxygen as Tri-Octyl Phosphine Oxide (TOPO) [Danilov, 2011].

Two basic dissolution procedures were tested: ammonia solution dissolves only WO_3, while a 10 % NaOH solution rapidly dissolves both WO_3 and $PbWO_4$. One mL of this solution is strongly acidified with 8 mL of 65 % nitric acid to promote the re-precipitation of WO_3 which has a monoclinic or orthorhombic structure [Lassner and Schubert, 1999; Wriedt, 1898], where all vertices are shared, and it does not bond Th and U. The precipitate is then centrifuged, the supernatant, containing Th and U, treated by TRU cartridges then it is analyzed. Recovery tests showed quantitative recovery of Th and U in the supernatant.

2.3.3. Ammonium paratungstate (APT) samples

APT samples were treated similarly to $PbWO_4$ and WO_3. Approximately 0.2 g of APT powder were dissolved in 10 % NaOH and then acidified with 9 mL of 65 % HNO_3. The same TRU resin-based matrix separation was applied. ICP-MS analysis was performed in both LR and MR modes, taking advantage of the lower interference levels in these matrices. The detection limits for Th and U were found to be below 1000 ng kg^{-1}.

2.4. Validation of the method

2.4.1. Conditioning of TRU resin cartridges

In addition to the use of ultrapure reagents and carefully cleaned labware, it is essential to minimize the background contribution from TRU resin cartridges. The cartridges were alternately rinsed with 5 mL of 4 M nitric acid and eluted with 10 mL of 0.1 M ammonium oxalate solution. The eluted solutions were analyzed by ICP-MS to monitor the Th and U levels until they became comparable to those of fresh solutions. As shown in Fig. 1, after four rinsing steps, the release of Th and U was found to be stable and acceptable for trace-level analysis.

2.4.2. Evaluation of procedural blanks and detection limits

Procedural blanks were evaluated to quantify the background contribution from reagents and resin. Across six tested columns, blanks remained low and consistent. In Table 1, detection limits (DLs) were calculated as three times the standard deviation of 20 blank measurements [European Commission Regulation No 333/2007] using 100 ng L^{-1} reference solutions to evaluate the instrumental sensitivity. For Pb samples, DLs for Th and U were approximately 0.5 ng kg^{-1} and 0.2 ng kg^{-1}, respectively considering a total dilution factor for the method ranges between 2 and 4. For tungsten-containing matrices the DL for Thorium, using MR mode to overcome the polyatomic interference, was around 50 ng kg^{-1} while the Uranium DL was 50 ng kg^{-1} in both MR and LR mode taking into account a dilution factor of about 300.

Although the signal intensity is reduced by a factor of 10 when using Medium Resolution mode, the performance of the method, in terms of detection limit, remains comparable, as it is primarily influenced by the background rather than instrumental sensitivity. This suggests that Medium Resolution (MR) is essential for matrices containing tungsten, particularly for thorium, where potential polyatomic isobaric interferences from $^{184}W^{16}O_3^+$ may occur, while still maintaining comparable sensitivity for uranium.

2.4.3. Evaluation of efficiency of the method

In a previous work, UTEVA resin was used for the preconcentration of Th and U in lead sample (Hoppe et al., 2015). Artificial isotopes of ^{229}Th and ^{233}U were then added directly to the sample to estimate the recovery of the natural isotopes, resulting in recoveries of 45 % for ^{229}Th and 12 % for ^{233}U. In principle, this approach allowed for a reliable measurement of Th and U, even though the recovery was relatively low and not fully reproducible across different columns. The authors attributed the reduced recoveries to the large rinse volume of nitric acid that was required to sufficiently reduce the remaining lead on the column. Other authors applied different procedures to elute Th and U separately from UTEVA resin, achieving recoveries of up to 60 % and 80 %, respectively (Grinberg et al., 2005). This study is based on the use TRU resin and the efficiency of the method was evaluated by adding known amounts of natural Th and U to six different aliquots of the original sample, since no artificial isotopes were available. The results, reported in Table 2, showed fully satisfactory recoveries for both thorium and uranium after eluting the resin with 10 mL of a 0.1 M solution of $(NH_4)_2C_2O_4$. This solution was directly analyzed by ICP-MS without significant matrix effects.

3. Results and discussion

3.1. Radiopurity characterization of archaeological lead

Using the developed analytical method, the Th and U contamination levels were measured in two Roman archaeological lead ingots and in powdered samples. Prior to analysis of the ingot samples, the outer surface was removed using 20 % nitric acid to eliminate any

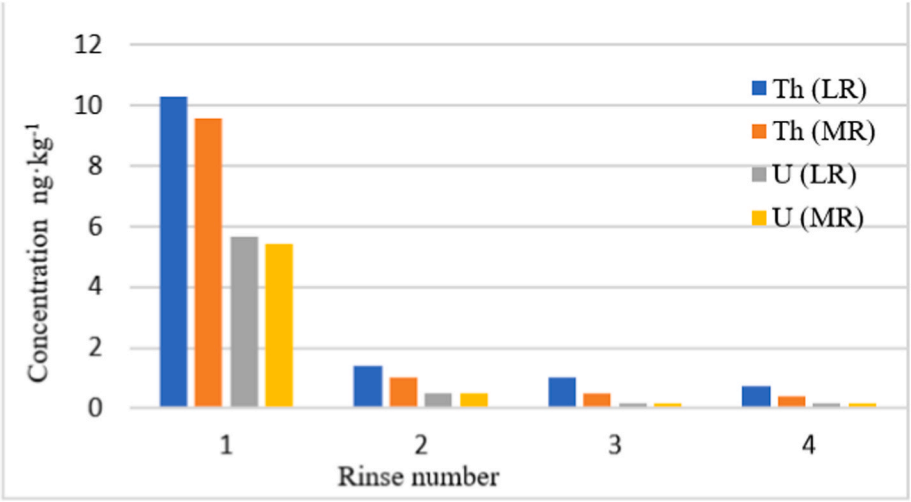

Fig. 1. Background equivalent concentration for Th and U during the conditioning of TRU resin cartridges. The data are the average values for six columns.

Table 1
Equivalent concentration of process blank and detection limit for lead and tungsten-based matrices.

	Average	ST Dev	DL for Pb	DL For W compound
	ng·kg^{-1}	ng·kg^{-1}	ng·kg^{-1}	ng·kg^{-1}
^{232}Th (LR)	0.50	0.05	0.5	N/A ($^{184}W^{16}O_3^+$)
^{232}Th (MR)	0.49	0.06	0.5	50
^{238}U (LR)	0.32	0.02	0.2	50
^{238}U (MR)	0.31	0.02	0.2	50

contamination introduced during handling and cutting. Multiple aliquots were prepared from each ingot for repeatability and recovery testing. Quantification was performed using an external calibration curve with Th and U concentrations in the range of 1–20 ng L^{-1} in 0.1 M ammonium oxalate solution. The results reported in Fig. 2 indicated good homogeneity within the same ingot, with Th and U levels averaging 1.2 ± 0.6 ng kg^{-1} and 1.2 ± 0.4 ng kg^{-1} respectively for ingot 2, and slightly higher values in ingot 1 (5.5 ± 1.6 ng kg^{-1} for Th and 9 ± 3 ng kg^{-1} for U).

Additionally, powdered archaeological lead samples were analyzed in two different laboratories (samples 7 and 8 at Milano-Bicocca; samples 9–11 at LNGS). Results showed consistent Th and U concentrations across replicates and sites, confirming the reproducibility and robustness of the method. Average concentrations from five replicates were 7.1 ± 1.5 ng kg^{-1} for Th and 4.1 ± 1.3 ng kg^{-1} for U.

3.2. Screening and characterization of tungsten oxides and crystals

A preliminary assay campaign was performed on WO_3 powders from different suppliers and on $PbWO_4$ powder and crystals synthesized from the most promising batch. Samples were dissolved in NaOH, diluted, neutralized, and analyzed via ICP-MS. Medium Resolution mode was essential for Th quantification to suppress isobaric interference. Although initial detection limits were ~1 ng g^{-1}, the screening allowed

Table 2
Recovery tests of ^{232}Th and ^{238}U from spiked archaeological lead samples. The combined standard uncertainty is reported with coverage factor k = 1.

		Rec Test 1	Rec Test 2	Rec Test 3	Rec Test 4	Rec Test 5	Rec Test 6	Average
Th	%	100 ± 6	98 ± 6	99 ± 5	96 ± 5	98 ± 5	95 ± 5	98 ± 2
U	%	101 ± 6	97 ± 6	98 ± 5	97 ± 5	98 ± 5	98 ± 5	98 ± 2

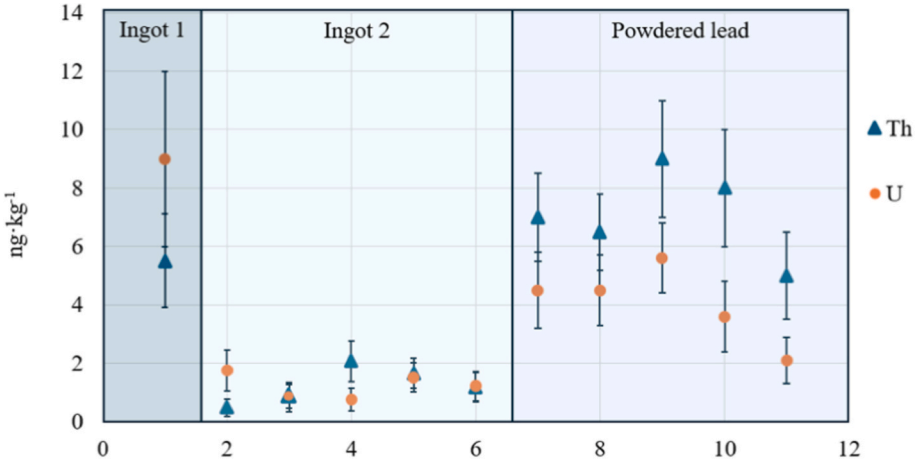

Fig. 2. Concentration of Th and U determined by ICP-MS in archaeological lead samples. The combined standard uncertainty is quoted with coverage factor k = 1.

the identification of the most radiopure WO_3 (type A) used for the first crystal synthesis. These last samples were analyzed one more time after the method development that significantly enhanced the DLs to 0.1 ng g^{-1} as shown in Fig. 3.

ICP-MS and gamma-ray spectrometry were used to analyze WO_3, $PbWO_4$ powder, and the final crystal. Type A WO_3 and derived powders and crystals showed low Th and U concentrations (<1 ng g^{-1}), while other WO_3 batches had U up to 3500 ng g^{-1}. The synthesis and growth processes did not introduce significant contamination, as verified by comparing the powder and final crystal results.

3.3. Analytical treatment and measurement of ammonium paratungstate (APT)

Since WO_3 has been identified as the starting material most affected by radioactive contamination, investigations are underway to explore alternative compounds for crystal production; ammonium paratungstate (APT) represents a promising candidate. The first APT samples were subjected to the same treatment procedure developed for WO_3 and $PbWO_4$. Around 0.2 g of APT powder was dissolved in 10 % NaOH and subsequently acidified with 9 mL of 65 % nitric acid. Following this, Th and U were separated from the matrix using TRU resin cartridges. ICP-MS analysis was performed in both Low and Medium Resolution modes. APT matrices showed lower levels of isobaric interference compared to WO_3, allowing quantification in LR mode as well. The detection limits achieved were <600 ng g^{-1} for Th (LR), <2000 ng g^{-1} for Th (MR), <800 ng g^{-1} for U (LR), and <2400 ng g^{-1} for U (MR). These results demonstrate the potential of the analytical method for evaluating commercial APT for use in low-background applications, but the DL should be improved by a factor 10 to match the RES-NOVA requirements.

4. Comparison between ICP-MS and gamma-ray spectrometry

To cross-validate the results obtained with ICP-MS, we performed gamma spectrometry analysis to assess the content of long-lived radionuclides in archaeological Pb. A sample of 97.366 kg was measured using an ultra-low background High-Purity Germanium (ULB-HPGe) detector, operated in the underground laboratories of LNGS [Laubenstein, 2017]. This approach provides a complementary and non-destructive method for evaluating the radiopurity of the Pb and serves as an essential step toward validating its use in ultra-sensitive detectors such as those envisioned for the RES-NOVA project.

While ICP-MS offers extremely low detection limits for trace elemental concentrations, it does not directly probe the radioactive isotopic composition or the secular equilibrium within decay chains. In contrast, gamma-ray spectrometry allows for direct identification and quantification of specific gamma-ray emitting radionuclides, enabling a more complete assessment of ^{238}U and ^{232} Th day chains. In particular, HPGe detectors provide the capability to test for secular equilibrium by identifying characteristic gamma-ray lines from Th and U progeny such as ^{228}Ra and ^{226}Ra, which could contribute to the background for the RES-NOVA experiment.

To improve the sensitivity of the measurement, special care was taken in the preparation of the Pb sample. The hundred kg of archaeological Pb was recast into brick-shaped ingots, which were stacked to maximize the geometric efficiency of the detector system.

The ULB-HPGe detector used for this study is the GeMPI-2 detector [G. Heusser et al., 2006] operated at the LNGS, Italy. This features an ultra-low background shielding system, including layers of radiopure copper and lead, as well as nitrogen flushing to suppress radon-induced backgrounds. The measurement lasted 68.36 d. The results of the 97.336 kg archaeological Pb screening are reported in Table 3. The activities for the different nuclides are estimated following the prescriptions of [Clemenza, 2012]. The activities of ^{232}Th and ^{238}U that were obtained on the basis of the measurements carried out by ICP-MS are also reported for comparison in parentheses. The good agreement of the two techniques demonstrates the substantial secular equilibrium of the decay chain of Thorium and Uranium.

With respect to tungsten oxide, ICP-MS measurements reported in Table 4 indicate that the parent nuclides of the uranium and thorium decay chains are present only below detection limits (<1 ng g^{-1}). In contrast, for the short-lived daughter nuclides particularly in samples

Table 3
Massic activity of radio nuclides measured by gamma-ray spectrometry in 97.336 kg of archaeological lead. The corresponding massic activity measured by ICP-MS for ^{232}Th and ^{238}U are reported in parentheses*.

Decay chain	Radionuclide	Massic activity [μBq·kg^{-1}]
^{232}Th (*24 ± 8)	^{228}Ac	40 ± 20
	^{212}Pb	160 ± 70
	^{212}Bi	<140
	^{208}Tl	<23
^{238}U (*100 ± 30)	^{214}Pb	40 ± 20
	^{214}Bi	<20
	^{235}U	<760
	^{40}K	<270
	^{137}Cs	<8.0
	^{207}Bi	<13
	^{202}Tl	60 ± 5

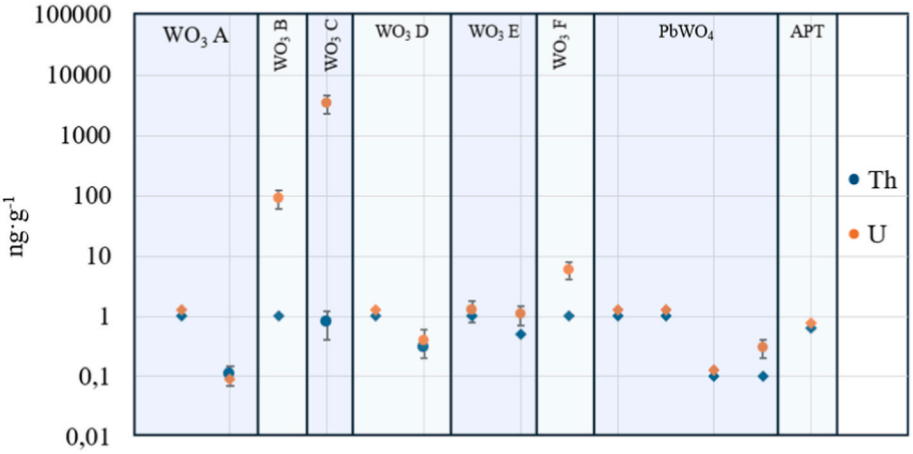

Fig. 3. Contamination in WO_3 and $PbWO_4$ samples. The data with rhombic markers are upper limits. For samples WO_3A_2, powder $PbWO_4$_3 and crystal $PbWO_4$_4 the newly developed method was applied.

Table 4
Contamination measured by gamma-ray spectrometry and ICP-MS* in WO_3 samples from three different suppliers. The combined standard uncertainty is quoted with coverage factor K = 1.

		WO_3 A [ng·g^{-1}]	WO_3 B [ng·g^{-1}]	WO_3 E [ng·g^{-1}]
^{232}Th		*0.11 ± 0.03	*<1	*<1
	^{228}Ra	<0.31	2.2 ± 0.5	18 ± 2
	^{228}Th	0.7 ± 0.1	2.5 ± 0.4	15 ± 1
^{238}U		*<0.1	*90	*1.2 ± 0.5
	^{234m}Pa		80 ± 10	
	^{226}Ra	0.08 ± 0.04	0.9 ± 0.1	15 ± 1

WO_3 E and WO_3 B significant contamination is observed, with concentrations well above 1 ng g^{-1} for nearly all radionuclides in both decay series.

The availability of simultaneous measurements of parent nuclides via ICP-MS and progeny radionuclides via gamma-ray spectrometry enables a clear identification of sample A as the cleanest among those analyzed. Consequently, this sample has been selected for use in the development and testing of archaeological lead tungstate ($PbWO_4$) crystal production.

These results strongly suggest that any initial secular equilibrium of uranium and thorium in the precursor materials has been disrupted, most likely due to chemical synthesis and purification processes involved in the manufacturing of tungsten oxide.

5. Conclusion

The radiopurity assessment of raw materials intended for the production of $PbWO_4$ crystals developed for the RES-NOVA experiment targeting astrophysical neutrino detection has confirmed the exceptional purity of archaeological lead, while identifying tungsten oxide (WO_3) as the primary source of potential radioactive contamination. Among the WO_3 samples analyzed, uranium concentrations varied by up to two orders of magnitude depending on the supplier, highlighting the importance of careful material selection.

To enable ultra-trace analysis of thorium and uranium, a preconcentration method based on selective chromatographic extraction resins, using ICP-MS as detector, was employed. This approach achieved matrix removal efficiencies of 99.95 % and spike recoveries approaching 100 %, with detection limits reaching 0.2 ng kg^{-1} for archaeological lead and 50 ng kg^{-1} for tungsten-containing samples. It was also observed that a significant portion of the procedural blank originates from ammonium oxalate used in the elution step. Consequently, detection limits could be further improved either by selecting higher-purity reagents or by reducing the elution volume to 5 mL, as preliminary results indicate recoveries remain above 90 %.

One hundred kg of archaeological lead matching the full raw material requirement for RES-NOVA were screened over 70 days using ULB-HPGe detector to search for short-lived radionuclides in the uranium and thorium decay chains. These measurements were used to assess potential disequilibrium. The complementary application of gamma-ray spectrometry and ICP-MS provides a robust multi-technique validation of the radiopurity of the materials, ensuring they meet the stringent requirements for crystal production.

For tungsten oxide specifically, ICP-MS data revealed that parent nuclides from the U and Th decay chains were consistently below the detection threshold (<1 ng g^{-1}). However, short-lived daughter isotopes particularly in samples WO_3 E and WO_3 B exhibited substantial contamination, exceeding 1 ng g^{-1} in nearly all cases. The combined use of ICP-MS and gamma-ray spectrometry enabled the identification of sample A as the cleanest tungsten oxide source. This material has therefore been selected for the production trials of archaeological lead tungstate crystals.

These results underline the importance of comprehensive radiopurity screening to ensure compliance with the low-background requirements of next-generation neutrino experiments.

CRediT authorship contribution statement

Luigi Cappelli: Writing – review & editing, Supervision, Funding acquisition. **Massimiliano Clemenza:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Data curation, Conceptualization. **Federico Filippini:** Writing – original draft, Methodology, Data curation, Conceptualization. **Matthias Laubenstein:** Writing – review & editing, Supervision, Formal analysis, Data curation, Conceptualization. **Stefano Nisi:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Lorenzo Pagnanini:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Luca Pattavina:** Writing – review & editing, Writing – original draft, Supervision, Funding acquisition, Conceptualization.

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Declaration of competing 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.

Data availability

Data will be made available on request.

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