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The quick and ultrasensitive determination of K in NaI using inductively coupled plasma mass spectrometry

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ABSTRACT

A highly sensitive, novel and quick assay method utilizing inductively coupled plasma mass spectrometry was developed for the determination of K in NaI powders and NaI(Tl) scintillator crystals for use in ultralow background applications. The determination of K (*viz.* ^{40}K), as well as Th and U and their daughters, is important in ultralow background detector materials to ensure incorporation of materials of sufficiently high radiopurity. Through the use of improved instrumentation, cool plasma operating conditions, and meticulously clean sample preparations, detection limits of 11 fg ^{nat}K g^{-1} (or 341 pBq ^{40}K kg^{-1}) was attained for K in pure water. Detection limits in the sample matrix (*i.e.*, NaI) were 0.529 ng ^{nat}K g NaI^{-1} (or 16.4 μBq ^{40}K kg NaI^{-1}). A number of different precursor NaI powder samples and NaI(Tl) scintillator crystals were assayed for their K content. Determinations ranged from 0.757 to 31.4 ng ^{nat}K g NaI^{-1}. This method allows for the screening of materials to unprecedented levels in a fraction of the time compared to gamma ray counting techniques, providing a useful method for a more effective screening tool of K in ultralow background detector materials.

1. Introduction

In next-generation rare-event physics experiments (*e.g.*, neutrinoless double beta decay detection [1–7], dark matter studies [8–23], and solar neutrino detection [24–26]), suppressing and accounting for background events is of great importance [27]. Some of the major contributors of background events are from naturally-occurring radiocontaminants (*e.g.,* ^{232}Th, ^{238}U and their daughters, *etc.*) present in minuscule quantities in detector components. Validation of the radiopurity of detector materials is required to ensure background specifications are obtained for optimal performance of the experiment at large. Due to the scale, complexity, and expense of modern experiments, it is imperative that the detector is not limited solely as a radioassay tool unto itself. The radiopurity required of most detector materials is well beyond levels typical of best industry practices. The stringent background requirements for next-generation detectors dictate that highly sensitive analytical assays are performed on all detector materials that could significantly contribute to the background.

Concerted efforts have been made for comprehensive assay and validation of detector materials [28–33]. There are a variety of quantitative (radio)assay techniques currently employed in the validation of detector materials. The three most common assay techniques used in ultralow background physics applications are gamma ray spectroscopy, nuclear activation analysis (NAA), and inductively coupled plasma mass spectrometry (ICP-MS).

Historically, at least until recently, gamma ray spectroscopy has been the most favored assay technique as it provides sensitive, non-destructive analyses in a manner that is relatively straightforward and well-known to the physics community. Gamma ray spectroscopy is particularly useful for assaying complicated, multi-component parts that can prove cumbersome for other assay techniques. However, to reach the ultralow levels (*e.g.,* 1 μBq ^{238}U/kg or 10 μBq ^{40}K/kg) oftentimes required for validation, gamma counting requires large amounts of material (*ca.* kg quantities) and very long counting times (*ca.* several months). Given that rare-event detectors are typically comprised of numerous parts, many of them very small, gamma ray spectroscopy can prove inadequate at effectively and efficiently screening them for their inclusion in the detector at the level required.

NAA overcomes the need for large samples and long counting times by effectively increasing the activity of the radiocontaminants through neutron bombardment. NAA analyses can be quite expensive and, depending on the material, produce undesirable radionuclides of high activity, resulting in long cool down times or cumbersome sample handling and oftentimes requiring chemical separation of radionuclides. For these reasons, NAA, while still providing niche applications for material assay, has fallen out of favor to gamma ray counting and ICP-MS methods.

ICP-MS is quickly becoming the favored technique for ultralow

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background materials assay. ICP-MS provides the sensitivity and high throughput for validating a wide variety of materials at μBq/kg (and lower) sensitivities for ^{232}Th and ^{238}U. Small samples (< 1 g) are typically required and analyses only take a day or two. Moreover, with recent advances in instrument design, ICP-MS methods can attain unprecedented detection limits for K, which was once extremely difficult due to problematic isobaric interferences at the same *m/z*. This study presents a test case for the ultrasensitive determination of K in detector materials, specifically NaI(Tl) crystals.

The determination and, more importantly, reduction of ^{40}K in detector materials is of particular interest to the ultralow background community. Collaborations such as ANAIS [34], DM-ICE [35], KIMS [36], and SABRE [37] look to further reduce backgrounds in NaI(Tl) based detectors in order to interrogate the significant and controversial claims by the DAMA/LIBRA collaboration of detecting signatures compatible with expections of WIMP dark matter interactions [38]. Limits set by other direct dark matter detection collaborations, such as SuperCDMS [39], XENON100 [40], and LUX [41] are in direct conflict with the DAMA/LIBRA claim. Much controversy stems from the *ca.* 3 keV signal that could stem from ^{40}K contamination in the crystal [42]. Collaborations employing independent NaI(Tl) crystals for the direct detection of dark matter can test the DAMA/LIBRA results [34–37]. Great efforts are underway to reduce the ^{40}K background in NaI crystals through a variety of means, such as, sourcing and/or producing the most radiopure starting reagents, attaining the most radiopure labware materials, and growing crystals using a process that does not further contaminate the crystal. The process is laborious, time-consuming, and requires validation through assay.

This study focuses on the development of an ICP-MS assay methodology for the relatively quick (< 1 day) and ultrasensitive determination of K (*viz.* ^{40}K) in ultralow background detector materials.

2. Experimental

2.1. Facilities

Laboratory studies were performed in a Class 10,000 cleanroom at Pacific Northwest National Laboratory (PNNL). A laminar flow hood providing a Class 10 environment was used for sample preparation.

2.2. Instrumentation

An Agilent 8800 s series ICP-MS (Agilent Technologies, Santa Clara, CA, USA) with an integrated autosampler was used for determination of K in precursor NaI and finalized NaI(Tl) scintillator crystals. The ICP-MS was equipped with a standard quartz double-pass spray chamber and a microflow nebulizer. Daily optimizations and tunings were performed and recorded. A 10% NH_3/90% He reaction gas of high purity (> 99.999%) was acquired from OXARC Incorporated (Spokane, WA, USA).

2.3. Reagents and labware

All chemicals and reagents were acquired at the highest purity from Fisher Scientific (Pittsburgh, PA, USA) unless otherwise noted. Ultralow background perfluoroalkoxy alkane (PFA) sample vials (Savillex, Eden Prairie, MN, USA), which also double as ICP-MS autosampler vials, were used as sample containers. As described previously [32], all labware was acid leached in 6 M HNO_3 and incubated in an 80 °C vacuum oven for at least 24 h. Vials were then removed and triply rinsed in deionized water. 1.5 mL of 5% HNO_3 (v/v) was deposited in each vial, capped, and again incubated in an 80 °C oven overnight before being analyzed for K content *via* ICP-MS. Vials passed validation when the measured counts were at background levels for reagent blank 5% HNO_3 (v/v) levels. Vials passing inspection were triply rinsed with deionized water before air drying in a Class 10 laminar flow hood with the vial cap loosely atop the vial. Vials that did not pass validation went through the leaching/validation procedure until they passed validation.

2.4. Quantitation and data analyses

A 1000 μg K mL^{-1} standard was used to make (gravimetrically) all stock and calibration standards (Inorganic Ventures, Christiansburg, VA, USA). The method of standard additions was employed for quantitation. Matrix-matched standards were created gravimetrically from stock solutions in a 1000 μg NaI mL^{-1} in 2% HNO_3 (v/v) matrix. Some samples had multiple replicates made from the stock sample in order to assess the variability from sample processing. Calibration and quantitation was performed using the signal at *m/z* 39; the ^{39}K isotope is significantly more abundant (93.26%) compared to ^{40}K (0.0117%) and ^{41}K (6.73%). Activities (μBq ^{40}K/kg NaI) were then calculated using the isotopic abundance and half-life ($t_{1/2}$=1.25×10^9 y) of ^{40}K. For reference, 1 ng ^{nat}K g^{-1} sample (*i.e.,* 1 ppb ^{nat}K) is equivalent to 31.0 μBq ^{40}K kg^{-1}.

2.5. Sample Origins

NaI precursor salts and finalized NaI(Tl) crystals were attained from two non-affiliated sources. Samples were provided by Alpha Spectra, Inc. (Grand Junction, CO, USA) and from Princeton University.

3. Results and discussion

3.1. Triple Quadrupole ICP-MS

The Agilent 8800 employs a “triple quadrupole” (QQQ) design. Analogous to instruments available for *molecular* mass spectrometry, the 8800-QQQ can operate in tandem MS mode (MS/MS). A collision/reaction cell (CRC) is in-line between the two quadrupoles, allowing ion/gas reactions to take place to remove the interference for analyte detection. Similarly, the CRC could be employed to shift the analyte (or internal standard) *m/z* to another mass range that is free from isobaric interferences. The QQQ design allows for myriad applications to improve analyte detection; the analysis of NaI for the determination of K is investigated herein. The Agilent application note for ^{39}K analysis of pristine waters was used as a guide in tuning the instrument for this study [43].

Instrument operating parameters such as cool plasma conditions and settings controlling the ion optics, mass analyzers, and CRC were tuned to maximize S/N for K while mitigating isobaric interferences. Historically, due to problematic polyatomic interferences, the sensitive detection limits required (*e.g.*, < ppb) for K analysis pertaining to ultralow background materials have been extremely challenging to achieve using ICP-MS. The determination of K is typically quantified using the signal at *m/z* 39, as it is the most abundant isotope (93.26%). However, all three isotopes of potassium (^{39}K$^+$, ^{40}K$^+$, and ^{41}K$^+$) exhibit significant isobaric interferences (^{38}ArH$^+$, ^{40}Ar$^+$, and ^{40}ArH$^+$, respectively) stemming from the Ar plasma/carrier gas. These are produced in great excess in a *ca.* 8000 K plasma operated at 1500–1600 W. These interferences make it difficult to obtain optimal detection limits. However, cool plasma conditions (600–800 W) can be used to remove much of the argon background while still providing a sufficiently energetic environment to ionize K, which has a relatively low first ionization potential (4.34 eV). Further, through the use of 10% NH_3/90% He reaction gas, the CRC can be employed to remove the surviving ^{38}ArH$^+$ background and a water gas cluster ion $H_3{}^{18}O(H_2{}^{16}O)^+$[or $H_3{}^{16}O(H_2{}^{18}O)^+$] at *m/z* 39 [43]. Through careful optimization and assiduously clean laboratory practices, an instrumental detection limit of 11 parts-*per-quadrillion* (fg K g^{-1}) was attained for K in pure water.

Table 1
K determinations in ng ^{nat}K g^{-1} and μBq ^{40}K kg^{-1}. The precision of each measurement is given as "± 1 stdev", which represents the standard deviation of triplicate measurements of the same sample. Some samples were prepared in duplicate or triplicate (Samples 8, 9, 10) to assess the variation in determined results from the sample processing.

Sample	ng ^{nat}K g^{-1}	± 1 stdev	μBq ^{40}K kg^{-1}	± 1 stdev
1	17.1	0.1	530	3
2	31.4	0.4	974	12
3	15.4	0.3	477	9
4	25.4	0.1	785	3
5	10.0	0.7	311	21
6	9.08	0.31	281	10
7	9.67	0.40	300	12
8_Rep_1	9.82	0.36	304	11
8_Rep_2	9.86	0.44	305	14
9_Rep_1	1.32	0.07	40.8	2.1
9_Rep_2	1.29	0.15	40.0	4.7
9_Rep_3	1.23	0.05	38.1	1.4
10_Rep_1	0.757	0.078	23.5	2.4
10_Rep_2	0.825	0.113	25.5	3.5
10_Rep_3	0.775	0.072	24.0	2.2

This instrument detection limit translates to 341 pBq ^{40}K kg^{-1} in pure water.

3.2. Method of standard additions and use of internal standard for the determination of K in NaI

In order to account for signal suppression due to introduction of a very concentrated solution (1000 μg NaI mL^{-1}), the method of standard additions was employed for accurate quantitation of K. All samples and calibration solutions were made up to a final matrix of 1000 μg NaI mL^{-1} in 2% (v/v) HNO_3. Through the employment of the QQQ, a sodium-ammonia gas adduct was used as an internal standard. The first quadrupole allowed the transmission of Na^+ into the CRC where ion-gas reactions can occur. NH_3 is extremely reactive gas and forms multiple sodium-ammonia adducts. The second quadrupole was then used to filter out the triply-substituted adduct (*viz.*, $Na(NH_3)_3^+$) at *m/z* 74. The analyte signal at *m/z* 39 was monitored in proportion to the internal standard signal at 74 *m/z*, allowing for corrections in signal variations due to instrument drift and plasma flicker (among others). Table 1 shows the determined results in ng ^{nat}K g^{-1} and μBq ^{40}K kg^{-1}. The standard addition calibration curve was very linear (R =0.99999). The detection limit (3× standard deviation of the blank) was determined to be 0.529 ng ^{nat}K g^{-1}, or 16.4 μBq ^{40}K kg^{-1}. All samples were determined above the detection limit.

Exact modifications to create a more radiopure crystal will not be discussed as it is beyond the scope of this assay capability study. However, these results were useful in efficiently determining the efficacy of changes made in order to reduce K contamination during the powder and crystal growth processes. In order to reach ultralow levels of K contamination within NaI(Tl) crystals, well-tailored and iterative approaches are necessary. To effectively assess the validity of method iterations and inform the next processing step, validation is required. This ICP-MS assay capability allows for the ultrasensitive validation of *ca.* 50 unique samples in a less than a day, whereas gamma ray counting methods would require very long counting times (*ca.* 3 months) and kg amounts of a *single* sample on an ultrasensitive high purity germanium detector to reach similar levels.

4. Conclusion

A powerful capability for the ultrasensitive determination of K in NaI has been described. Through the use of ultraclean sample processing, cool plasma operating conditions, and the triple quadrupole ICP-MS, extremely sensitive and relatively quick assays of materials can be realized. Detection limits ranged from the fg ^{nat}K g^{-1} range in ideal matrices (*e.g.*, water) to sub-ng ^{nat}K g^{-1} levels within a solid (*e.g.*, NaI). This capability will find continued use in the ultralow background community. Future assays will explore the determination of K in other common ultralow background detector materials, *e.g.*, copper, polymers, *etc.* Moreover, as this study provided just one example of the power of employing a triple quadrupole, its utility will be explored to further improve sample analyses of a variety of analytes in other arenas.

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