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A comparative study of ^{129}I content in environmental standard materials IAEA-375, NIST SRM 4354 and NIST SRM 4357 by Thermal Ionization Mass Spectrometry and Accelerator Mass Spectrometry

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Abstract

Iodine environmental measurements have consistently been validated in the literature using the standard material IAEA-375, soil collected approximately 160 miles northeast of Chernobyl, which is currently the only soil/sediment material with a certified ^{129}I activity. IAEA-375 has not been available for purchase since approximately 2010. Two other standard materials that are available (NIST SRM 4354, freshwater lake sediment and NIST SRM 4357, ocean sediment) have certified activities for a variety of radionuclides but not for ^{129}I. This paper reports a comparison of TIMS and AMS data for all three standards.

Keywords: AMS; TIMS; iodine; ^{129}I; standards

1. Introduction

The long-lived radioactive isotope ^{129}I with a half-life of 15.7 million years is produced naturally by cosmic ray spallation of xenon in the atmosphere and spontaneous fission of ^{238}U. However, the majority of the ^{129}I inventory in the environment is from anthropogenic activities associated with nuclear fuel reprocessing and to lesser extents from nuclear weapons testing and nuclear accidents such as Chernobyl and Fukushima (Povinec, et al., 2013; Schmidt, et al., 1998). While

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^{129}I is radioactive and radiometric techniques can be applied, the half-life is sufficiently long that mass spectrometry is more sensitive for ^{129}I quantification. Pre-nuclear materials have $^{129/127}$I ratios from 0.04 – 3 × 10^{-12} (Schmidt, et al., 1998; Daraoui and Riebe, 2016) and the most sensitive method capable of making such extreme isotopic ratio measurements is Accelerator Mass Spectrometry (AMS).

Due to its long half-life, ^{129}I has proven to be very useful as a tracer for environmental processes and age dating up to 80 million years (Fabryka-Martin, et al., 1984; Liu, 2015) leading to a growing number of AMS laboratories around the world analyzing ^{129}I. Along with this surge in AMS laboratories interested in ^{129}I comes an increase in the demand for readily available standard materials in order to verify analytical methods. Iodine environmental measurements have consistently been validated in the literature by using the standard material IAEA-375, soil from Novozybkov, Russia, which is a city roughly 160 miles northeast of Chernobyl, which is the only soil/sediment material with a certified ^{129}I activity. However, IAEA-375 has not been available for purchase since approximately 2010. NIST produced SRM 3230 and 3231, iodine isotopic standards at four different $^{129/127}$I ratios ranging from 10^{-12} to 10^{-6}. Although SRM 3230 and 3231 were very useful for their intended purpose, they do not represent an environmental matrix and are no longer available. A number of other standards have been either certified or analyzed for ^{129}I concentration; however, most of these are also unavailable (Zhang and Hou, 2013).

Two other environmental standard materials are available (NIST SRM 4354, freshwater lake sediment and NIST SRM 4357, ocean sediment) and have certified activities for a variety of radionuclides but not for ^{129}I. Previous work has shown that ^{129}I is present in these materials

(Qiao, et al., 2002; Izmer, et al., 2004); however the data available in the literature have been insufficient to establish a consensus value for ^{129}I. These materials have been analyzed at INL using two chemical separation and mass spectrometric analysis techniques. They involve a combustion method of the starting material followed by thermal ionization mass spectrometry (TIMS) analysis, and an aqueous leaching preparation for analysis by accelerator mass spectrometry (AMS). Both methods were validated via analyses of IAEA-375 for ^{129}I and show good agreement with the certified activity of 1.7×10^{-3} Bq kg^{-1} for ^{129}I; (1.8×10^{-3} Bq kg^{-1} by TIMS and 1.6×10^{-3} Bq kg^{-1} by AMS) with both sets of results within the 95% confidence interval of the certified value.

2. Experimental

2.1 Preparation of samples for TIMS

The combustion method that was used to process the soil samples was adapted from a technique developed for neutron activation analysis. Aliquots of each standard were spiked with ^{125}I, loaded into combustion tubes and heated at 850 ^{o}C in an oxygen atmosphere for one hour. It has been shown that the majority of the iodine is volatilized from soil samples at temperatures above 700 ^{o}C (Muramatsu, et al. 2008). This initial step equilibrates the ^{125}I with the sample as the combustion products transfer to a charcoal trap. The charcoal trap was then heated under nitrogen gas flow to pass the sample through quartz wool and onto a hydrated manganese dioxide (HMD) trap. The iodine was further purified chromatographically through the HMD with oxygen gas flow and at 600 ^{o}C to remove additional halides, such as, bromine and chlorine. The sample was collected back on a small volume of charcoal which was subsequently heated under nitrogen to drive the iodine into an ammonium hydroxide solution. The chemical yield of the process was measured by gamma assay of the ^{125}I in the trapping solution (Horrocks, 1975; Herod, 2014). The sample was loaded onto a rhenium ribbon by in situ precipitation of silver iodide using silver nitrate for analysis by thermal ionization mass spectrometry using a triple filament technique (Delmore, 1982).

2.2 Preparation of samples for AMS

Aliquots of each standard were leached in a 5% tetramethylammonium hydroxide (TMAH) solution at approximately 90 ^{o}C for over twelve hours. The size of aliquot leached was determined by the expected total iodine concentration in the standard. Approximately 1 – 2 grams of each standard was leached in 30 – 75 mL of 5% TMAH. Either a heated ultrasonic or reciprocating bath was used to aid the leach. An aliquot of each leachate was subsequently diluted for ^{127}I analysis by ICP-MS.

Iodine carrier (Inorganic Ventures Ion Chromatography Standard (ICI1) with an ~$^{129/127}$I ratio < 2×10^{-13} and certified iodide concentration of 1.002 μg mL^{-1} was added to adjust the $^{129/127}$I ratio to approximately 5×10^{-11} for the AMS analysis of ^{129}I. To ensure isotopic equilibration between the leachate and carrier, samples were taken through a series of redox steps (Glendenin and Metcalf, 2009). In brief, during the redox treatment, all iodine species present are oxidized to iodate/periodate, reduced to iodine and collected in cyclohexane. The iodine is oxidized to iodide and extracted into a dilute aqueous sodium bisulfite solution and then the organic layer was discarded. Aliquots of the sodium bisulfite solution were added to an electrochemical cell where the iodide was deposited for two hours on a silver cladded niobium wire working electrode. The wire and silver iodide deposit were pressed into a titanium cathode for ^{129}I analysis by AMS.

2.3 Measurement by TIMS

TIMS measurements were performed on a triple sector instrument constructed around the VG Sector 54 platform using the VG source housing, two VG magnets and an electrostatic analyzer

(ESA) in a BBE-C configuration. The overall design is described elsewhere (Stoffel and Laue, 1991) and has an abundance sensitivity of ~ 10^{11}. The instrument was installed at INL in 2002 and after some modifications has been used for iodine analysis since 2003. Samples are loaded as silver iodide on a side rhenium filament positioned in close proximity to a center rhenium filament that is coated with lanthanum hexaboride in a triple-filament arrangement (Delmore, 1982). In order to reduce the hydride interferences, negative TIMS is used to analyze the iodide anions at an energy of 6 keV. Following the first magnetic sector are two Faraday cups, one on the high mass side and one on the low mass side, positioned to collect the abundant ^{127}I isotope while allowing the minor ^{129}I and ^{125}I isotopes to pass through the last two sectors. ^{129}I and ^{125}I are counted on a discrete dynode electron multiplier located after the ESA. Alternating simultaneous measurements of $^{125/127}$I then $^{129/127}$I are performed during an analysis. The temperature of the sample filament is slowly ramped during an analysis to allow data collection at varying signal intensities. After plotting the signal intensities of the different isotopes against each other a linear regression is applied to the data and the slope of the ^{129}I intensity vs the ^{127}I intensity represents the $^{129/127}$I ratio; conversely for the $^{125/127}$I ratio. The ^{125}I is used to determine the yield through the combustion chemistry as well as quantify the ^{129}I in the TIMS measurement.

Quality control samples included with the TIMS samples are process blanks, NIST SRM 3231 ($^{129/127}$I = 0.982 (± 0.012) × 10^{-8}) standards and Blank Iodine Isotopic Standards ($^{129/127}$I = 16 (± 5) × 10^{-15}). Cross calibration of the detectors is performed using a technique developed at INL which uses the minor oxygen isotopes of ReO_4^- to simultaneously calibrate the discrete dynode with the Faraday cups. Essentially, the major isotope at mass 251 is measured on a Faraday cup located after the first magnetic sector and the minor isotope at mass 255 is measured on the discrete dynode simultaneously (theoretical 255/251 = 2.52×10^{-5}).

2.4 Measurement by AMS

The instrument used to make the AMS measurements is an NEC 0.5 MV compact accelerator mass spectrometer designed to analyze iodine in the +3 or the +2 charge state. This instrument consists of a low energy injection magnet followed by the accelerator and then a high energy analyzing magnet followed by an ESA. The low and high energy sides of the instrument have magnetic bouncing systems to rapidly switch the isotopes by changing the energy of the ions as they pass through the magnetic fields of the injection and analyzing magnets. Samples are loaded as solid silver iodide that has been electrodeposited on silver cladded niobium wire using the same procedure reported by our lab for electrodepostion on silver wire (Adamic, et al., 2015) and pressed into titanium cathodes for targets in the NEC SNICS source. The SNICS source produces negative ions by cesium sputtering and the iodide anions are selected for analysis in the instrument. The negative ions are accelerated into a stripper cell at the center of the accelerator (0.5 MV) where they collide with the stripper gas (helium or argon) and undergo a charge reversal. The energetic process of stripping electrons from the ions eliminates most molecular interferences from the spectrum. Iodine in the +2 charge state is normally selected for the analysis resulting in a transmission of 40% through the accelerator using helium as a stripper gas. The isotopic measurement is performed by rapidly switching (~7 Hz) the $^{127}I^{+2}$ into an offset Faraday cup located after the high energy analyzing magnet and then switching the $^{129}I^{+2}$ into the gas filled ionization detector located after the ESA.

A series of quality control samples interleaved amongst the samples are loaded on the 40 position sample wheel to correct for background and normalize the data to cross calibrate the detectors. The QC samples include matrix blanks, matrix blanks exposed to the sodium hydroxide electrodeposition, carrier blanks and INL-C normalization standards ($^{129/127}$I = 5.62 (± 0.03) × 10^{-11} *(k=1)* calibrated using NIST SRM 3230).

3. Results and discussion

3.1 Reference materials

IAEA-375

IAEA-375 (soil contaminated with Chernobyl fallout) has a total iodine concentration of approximately 1.4 $\mu g_{(I)}$ g^{-1} soil and certified ^{129}I value of 1.7 (± 0.4) × 10^{-3} Bq kg^{-1}, and recently has been measured to be 1.59 (± 0.08) × 10^{-3} Bq kg^{-1} (Jiang, et al., 2005). IAEA-375 has not been available for purchase since approximately 2010. Being the only available soil/sediment material with a certified ^{129}I concentration, it was used to validate the two methods reported in this paper. The certificate recommends 6 g aliquots for homogeneity of radionuclides, however, due to the limited supply of material smaller aliquots were chosen based on the estimated total iodine needed for an assay. Ten, one gram aliquots of the standard were leached in 50 mL of 5% TMAH. The AMS results obtained are summarized in Table 1.

Table 1. IAEA-375 AMS Results

IAEA 375	**^{129}I/^{127}I**	**% uncertainty *(k=1)***	**^{129}I, Bq kg^{-1}**	**% uncertainty *(k=1)***
1	1.81×10^{-7}	2.75%	1.51×10^{-3}	2.81%
2	1.96×10^{-7}	2.03%	1.64×10^{-3}	2.10%
3	1.89×10^{-7}	1.21%	1.61×10^{-3}	1.33%
4	1.89×10^{-7}	0.56%	1.63×10^{-3}	1.04%
5	1.98×10^{-7}	2.53%	1.55×10^{-3}	2.68%
6	1.91×10^{-7}	6.51%	1.60×10^{-3}	6.57%
7	1.90×10^{-7}	0.36%	1.61×10^{-3}	0.95%
8	1.82×10^{-7}	1.13%	1.61×10^{-3}	1.43%
9	1.91×10^{-7}	1.02%	1.63×10^{-3}	1.35%
10	1.88×10^{-7}	0.86%	1.59×10^{-3}	1.23%

IAEA 375	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I, Bq kg^{-1}**	**% uncertainty *(k=1)***
Average	**1.89×10^{-7}**	**2.77%**	**1.60×10^{-3}**	**2.52%**

Two, one gram aliquots of the standard were prepared for TIMS. The data obtained are summarized in Table 2.

Table 2. IAEA-375 TIMS Results

IAEA 375	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**Bq kg^{-1}**	**% uncertainty *(k=1)***
1	1.58×10^{-7}	1.58%	1.78×10^{-3}	1.99%
2	1.64×10^{-7}	0.82%	1.82×10^{-3}	1.47%
Average	**1.61×10^{-7}**	**2.50%**	**1.80×10^{-3}**	**1.57%**

Table 3 shows the good agreement of the TIMS and AMS measurements with the literature and the certified value for IAEA-375 demonstrating the validity of the two methods for quantifying ^{129}I in the environmental soil samples.

Table 3. Comparison of TIMS, AMS, literature and IAEA-375 certificate

IAEA 375	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I Bq kg^{-1}**	**% uncertainty *(k=1)***
TIMS	1.61×10^{-7}	2.50%	1.80×10^{-3}	1.57%
AMS	1.89×10^{-7}	2.77%	1.60×10^{-3}	2.52%
(Jiang, et al., 2005)	n/a	n/a	1.59×10^{-3}	2.52%
Certified	n/a	n/a	1.70×10^{-3}	11.76%

NIST SRM 4354 and NIST SRM 4357

Two standard materials that are available (NIST SRM 4354, freshwater lake sediment and NIST SRM 4357, ocean sediment) have certified activities for a variety of radionuclides but not for ^{129}I. Previous literature studies (Qiao, 2012; Izmer, 2004) have shown that ^{129}I is present in these materials; however, the data available in the literature have been insufficient to establish a consensus value for ^{129}I.

The NIST SRM 4354 (Freshwater Lake Sediment) has a total iodine concentration of approximately 7 $\mu g_{(I)}$ g^{-1} soil and literature ^{129}I value of 4.4 (± 0.6) × 10^{-2} Bq kg^{-1} (Qiao, 2012). Ten, one gram aliquots of NIST SRM 4354 were leached in 75 mL of 5% TMAH. The data obtained are summarized in Table 4.

Table 4. NIST SRM 4354 AMS Results

NIST SRM 4354	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I, Bq kg^{-1}**	**% uncertainty *(k=1)***
1	1.93×10^{-6}	2.09%	7.88×10^{-2}	1.77%
2	1.84×10^{-6}	3.46%	7.31×10-2	1.85%
3	1.83×10^{-6}	1.67%	7.46×10^{-2}	1.65%
4	1.90×10^{-6}	1.80%	7.68×10^{-2}	1.57%
5	1.95×10^{-6}	8.40%	7.84×10^{-2}	1.49%
6	1.93×10^{-6}	2.33%	7.75×10^{-2}	1.73%
7	1.92×10^{-6}	1.82%	7.93×10^{-2}	1.67%
8	1.95×10^{-6}	2.24%	7.84×10^{-2}	1.61%
9	2.03×10^{-6}	2.47%	7.65×10^{-2}	1.55%
10	1.99×10^{-6}	6.80%	8.04×10^{-2}	1.57%
Average	**1.93×10^{-6}**	**3.19%**	**7.74×10^{-2}**	**2.85%**

Two, one gram aliquots of NIST SRM 4354 were prepared for TIMS. The data obtained are summarized in Table 5.

Table 5. NIST SRM 4354 TIMS Results

NIST SRM 4354	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I, Bq** kg^{-1}	**% uncertainty *(k=1)***
1	1.97×10^{-6}	0.72%	8.10×10^{-2}	1.03%
2	1.92×10^{-6}	1.43%	8.90×10^{-2}	1.24%
Average	**1.94×10^{-6}**	**1.56%**	**8.50×10^{-2}**	**6.65%**

The two mass spectrometric techniques applied to NIST SRM 4354 in this work show agreement with each other and when combined with the results from IAEA-375 leads to a high degree of confidence in the data. However, as can be seen in Table 6 the results do not agree with the previously reported value in the literature (Qiao, et al., 2012). The source of this discrepancy is unknown and warrants further measurements by additional laboratories to form a consensus value that could be useful to the AMS community.

Table 6. Comparison of TIMS, AMS and NIST SRM 4354 literature value (Qiao, et al., 2012)

NIST SRM 4354	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I Bq kg^{-1}**	**% uncertainty *(k=1)***
TIMS	1.94×10^{-6}	1.56%	8.50×10^{-2}	6.65%
AMS	1.93×10^{-6}	3.19%	7.74×10^{-2}	2.85%
Literature	9.28×10^{-7}	1.83%	4.36×10^{-2}	13.88%

The NIST SRM 4357 (a blend of material collected off the coast of Sellafield, UK and in the Chesapeake Bay, USA in the weight ratio of 1:200, respectively) has a total iodine concentration of approximately 3.1 $\mu g_{(I)}$ g^{-1} soil and uncertified ^{129}I value of approximately 9×10^{-3} Bq kg^{-1}. Twelve, 1.5 - 2 gram aliquots of NIST SRM 4357 were leached in approximately 30 mL of 5% TMAH. The data obtained are summarized in Table 7.

Table 7. NIST SRM 4357 AMS Results

NIST SRM 4357	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I Bq kg^{-1}**	**% uncertainty *(k=1)***
1	5.73×10^{-8}	3.19%	1.18×10^{-3}	2.49%
2	6.19×10^{-8}	3.61%	1.25×10^{-3}	3.01%
3	5.52×10^{-8}	3.21%	1.26×10^{-3}	2.51%
4	6.29×10^{-8}	3.23%	1.39×10^{-3}	2.54%
5	4.77×10^{-8}	3.11%	1.09×10^{-3}	2.39%
6	4.64×10^{-8}	3.26%	1.06×10^{-3}	2.58%
7	4.72×10^{-8}	3.18%	1.10×10^{-3}	2.48%
8	4.83×10^{-8}	3.52%	1.09×10^{-3}	2.90%
9	3.06×10^{-8}	3.17%	1.06×10^{-3}	2.46%
10	4.45×10^{-8}	4.32%	1.03×10^{-3}	3.83%
11	4.62×10^{-8}	3.26%	1.10×10^{-3}	2.57%
12	3.69×10^{-8}	3.89%	1.08×10^{-3}	3.34%
Average	**4.88×10^{-8}**	**19.45%**	**1.14×10^{-3}**	**9.31%**

Two, one gram aliquots of NIST SRM 4357 were prepared for TIMS. The data obtained are summarized in Table 8.

Table 8. NIST SRM 4357 TIMS Results

NIST SRM 4357	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I Bq kg^{-1}**	**% uncertainty *(k=1)***
1	4.03×10^{-8}	2.52%	1.02×10^{-3}	1.68%
2	4.30×10^{-8}	3.30%	1.07×10^{-3}	2.05%
Average	**4.16×10^{-8}**	**4.52%**	**1.05×10^{-3}**	**2.81%**

The certificate for NIST SRM 4357 lists an uncertified value for the ^{129}I concentration (9×10^{-3} Bq kg^{-1}). The TIMS and AMS values are shown in comparison to this value in table 9. Again, there is a large inconsistency in the reported values. As was the case with NIST 4354, the ^{129}I concentration in NIST 4357 should be assessed by multiple laboratories in an effort to get a useful value that can be used by the community.

Table 9. Comparison of TIMS, AMS and NIST SRM 4357 uncertified value

NIST SRM 4357	**$^{129}I/^{127}I$**	**% uncertainty *(k=1)***	**^{129}I Bq kg^{-1}**	**% uncertainty *(k=1)***
TIMS	4.16×10^{-8}	4.52%	1.05×10^{-3}	2.81%
AMS	4.88×10^{-8}	19.45%	1.14×10^{-3}	9.31%
Uncertified	n/a	n/a	9×10^{-3}	n/a

4. Conclusions

The ^{129}I activity and $^{129/127}$I ratio have been quantified in three different reference materials: IAEA-375 (soil collected approximately 160 miles northeast of Chernobyl), NIST SRM 4354 (freshwater lake sediment) and NIST SRM 4357 (ocean sediment) by two independent chemical and mass spectrometric measurement techniques, AMS and TIMS. Both of the methods were validated using IAEA-375 which is the only soil/sediment material with a certified ^{129}I activity. Results from TIMS (1.8×10^{-3} Bq kg^{-1} ± 1.57%) and AMS (1.6×10^{-3} Bq kg^{-1} ± 2.52%) agreed very well with the IAEA-375 certified value (1.7×10^{-3} Bq kg^{-1}) for ^{129}I and both were within the 95% confidence interval on the certificate. The fact that IAEA-375 has not been available for purchase since approximately 2010 necessitates further characterization of other reference materials for ^{129}I. Two other environmental standard materials that are available NIST SRM 4354 and NIST SRM 4357 have certified activities for a variety of radionuclides but not for ^{129}I. The large uncertainty associated with the AMS $^{129/127}$I ratios for NIST SRM 4357 point to some heterogeneity issues with the smaller aliquot sizes used at INL. It is also possible that NIST SRM 4357 presented some matrix effects that influenced the ICP-MS results that are used to calculate the AMS $^{129/127}$I ratios. Although the AMS and TIMS measurements agreed reasonably well in this work, the discrepancy between those values and what could be found in the literature for NIST SRM 4354 and 4357 suggest that a more thorough ^{129}I assessment needs to be done, including other laboratories, in order to be able to use these two NIST standards for a quality control sample in the analysis of iodine. Clearly, there is an immediate need in the AMS community for certified reference materials with known ^{129}I concentrations.

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