

**USACE FUSRAP Maywood Team Identifies Challenges and Initiates  
Alternate Solutions Relating to the Radiochemical Analysis of Borosilicate  
Fiber Filters for Isotopes of Uranium and Thorium**

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**ABSTRACT**

This presentation discusses the primary purposes of particulate radionuclide air monitoring at the US Army Corps of Engineers (USACE) Formerly Utilized Sites Remediation Program (FUSRAP) Maywood Superfund Site (FMSS), the challenges encountered by the team when standard radiochemistry analytical methods are attempted on borosilicate fiber filter samples, the surrogate evaluations used when sample specific isotopic analysis is unsuccessful, and current strategies for overcoming radiochemistry method deficiencies. Typical borosilicate fiber filter sample preparation procedures including tracer spike and digestion methods and their impact on uranium and thorium data quality are of particular interest. Analytes discussed include isotopic uranium (U-234, U-235, and U-238) and isotopic thorium (Th-228, Th-230, and Th-232). Efforts to obtain reproducible and defensible results also included discussions with commercial laboratory radiochemistry managers as well as industry experts. This presentation may benefit sites that use similar sample collection and analysis techniques, utilize data that may have unidentified method-related issues with diminished data quality, or have a similar isotopic signature.

**INTRODUCTION**

The FMSS sites include various properties that contain elevated levels of Naturally Occurring Radioactive Material (NORM) generated from chemical processing of monazite sands during the first half of the twentieth century. Monazite sands are naturally-occurring ores that contain thorium oxide, typically at levels between 3% and 9% and to a much lesser extent uranium oxides. The spent monazite sands in the process waste were placed in man-made lagoons at the plant site. Over the years, storm water events have transported the contaminated waste over a large area covering several square miles. The contamination has been characterized and

remediation has been taking place over approximately the past 20 years. Radiation workers and members of the public in the vicinity of the Maywood sites during remediation activities may receive internal radiation exposure from inhalation of suspended dust particulates that contain NORM. Collection of airborne particulates is conducted at the FUSRAP Maywood Superfund site to characterize potential inhalation exposures to site workers and the general public. There are three types of air filter samples collected.

breathing zone samples;  
work area samples  
environmental area (perimeter) samples

The breathing zone, general area and perimeter filters are analyzed for gross alpha. The gross alpha results obtained must be converted to a dose, therefore, the isotopic signature of the collected particulates must be determined. This is done either based upon average activities of data from soil samples from remediation areas, or the isotopic signature must be determined by analysis. This paper focuses on the efforts employed to analyze perimeter filters for isotopic thorium and isotopic uranium using various acid leaching and digestion techniques, the surrogate evaluations used when analysis is unsuccessful, and strategies for employing alternate radiochemistry methods.

### **BOROSILICATE FIBER FILTER SAMPLE PREPARATION – CHALLENGES**

There are two options which may be used to convert the soil particulates on a borosilicate air filter to a form that can be measured for specific isotopes using radioanalytical instrumentation. These include leaching and total digestion of particulates and filter. Alternatively, the filter may be measured directly using gamma spectrometry. Each of these options shall be discussed separately.

Prior to leaching and total digestion, it is often advantageous to grind and/or muffle air filter samples. Grinding improves both leaching and digestion because it exposes a larger surface area to the leaching or digestion agents. For a given weight of solid material, the smaller the particle size, the greater the surface area. Heating the filters in a muffle furnace at temperatures between 400 and 650°C for a period (typically) of several hours destroys most organic compounds which might otherwise interfere with the sample preparation. Air filters were not ground prior to digestion or leaching.

#### **Direct Measurement**

Direct measurement involves measurement of the radionuclides of interest using a direct method such as gamma spectroscopy. The Maywood perimeter filters are rectangular, with dimensions of 20.3 cm x 27.9 cm (8 inch X 10 inch), so a circle of between 1.25 cm and 3.81 cm (0.5 inch and 1.5 inch) diameter (the actual diameter would be maximized so that it was close to the cross-sectional area of the gamma spectrometer's germanium crystal detector) would have to be cut out of the 20.3 cm x 27.9 cm filter. The circular filter cutout would represent a fraction of the overall filter, and that fraction would equal the fraction of the volume of air passed through the filter. For example, for a 2.54 cm (one inch) diameter circular cutout, the area of the cutout is

approximately 1% of the overall area of the filter. Thus, the activity of the cutout would be divided by 0.01 to obtain the total activity on the filter. The efficiency of the gamma spectrometer typically falls between about 0.2% to 3%, with most gamma energies having detector efficiencies less than 1%. Therefore, the primary drawbacks to direct measurement by gamma spectroscopy compared to leaching or digestion of the filter followed by alpha spectroscopy, is low efficiency (the alpha spectrometer has a much higher efficiency, typically 21-23%) and smaller filter aliquot size (for the digestion or leaching/alpha spectroscopy method the aliquot would be one quarter or one-fifth of the overall filter (for measurement of isotopic uranium, isotopic thorium, and radium-226)). In addition, alpha spectroscopy is capable of detecting more radionuclides than gamma spectroscopy. Non-homogeneity of the deposited solid is also a problem if one is selecting an aliquot of the sample for measurement since the aliquot may not be representative of the overall filter. This problem would be more severe for gamma spectroscopy since the aliquot is so much smaller.

### **Leaching**

Leaching is the process whereby a solvent is used to solubilize/remove the analyte of interest from the filter media in a quantitative or predictable manner. For borosilicate filters with dust particulates, some type of acid leach would be used to remove a given radionuclide. The main advantage of leaching is the short duration. The leaching process may take a couple of hours or less, whereas a digestion could take up to a day or more. The degree of leaching will vary depending upon the number of times the filter is flushed with the leaching solution, the type of leaching solution and its concentration, the contact time of the solution with the filter, the type of analyte and the chemical species within which it exists, and the analyte matrix, including particle size, density, and whether the analyte of interest is chemically absorbed or physically adsorbed.

Leaching of a borosilicate filter can be problematic because the laboratory may have difficulty determining the degree of leaching. The primary reason for this is the difference in the elemental species found in a typical standard versus that found in an environmental sample. For example, if we consider isotopic thorium analysis by alpha spectroscopy, the thorium-230 (Th-230) and thorium-229 (Th-229) standards are provided in a dilute nitric acid solution. When this standard and tracer are added to a filter, one would expect the standard or tracer to be quantitatively leached, since it exists in a completely soluble form. The thorium that may exist in dust particulates will likely be in the form of thorium oxide, the leachability of which is more difficult to predict. Standards for this compound would have to closely mimic the soil particulate type and size distribution. To the best of our knowledge, such standards are not available, probably because they would be so expensive as to be economically and practically unfeasible.

Some leaching tests were conducted in February and March 2006 at the US Army Corps of Engineers FUSRAP Maywood Laboratory (UFML). The results are described in the Discussion section.

### **Total Digestion of Particulates and Filter**

This paper will focus primarily on the total digestion technique. The advantages of total digestion are that the analytes of interest are all in a soluble form. Radioactive standards called

tracers can then be used to predict the recovery of the analytes of interest. In order to achieve this goal, both the dust particulates as well as the borosilicate air filter must be dissolved. Borosilicate is typically dissolved using hydrofluoric acid. Dust and soil particulates may contain organic material, metal oxides, silicates and insoluble metal salts. Organic material can be thoroughly oxidized with an oxidizing agent such as concentrated nitric acid, hot concentrated sulfuric acid, or hydrogen peroxide. Metal oxides can typically be broken down with concentrated acids. Typically a mixture of concentrated hydrochloric acid and concentrated nitric acid will dissolve most metal oxides. Refractory oxides may be more difficult to dissolve and may therefore require alternative sample preparation techniques.

## **DISCUSSION**

### **Air Filter Selection**

For the purposes of air sample collection, a filter must have the physical durability to be able to maintain its collection efficiency over extended periods of time (weeks) at high flow rates. For example, site perimeter samples are collected continuously at a flow rate of 13 cubic meters per minute, over a several week period of weeks in order to achieve a measurement sensitivity of approximately ten percent of the site's derived effluent limit (DEL) value for each primary radionuclide of concern. The DEL is equivalent to a dose equivalent of 0.1 millisieverts per year to members of the public from site particulate radionuclide emissions.

Evaluation of a series of filter media types and type-specific variants were conducted at the FMSS to determine which type could best meet the sampling needs. With the exception of borosilicate fiber filters in an eight by ten inch filter size, no other filter media tested could withstand the high flow rates, high sample loading over time, high collection efficiency requirements, and changing outdoor environmental conditions without filter failure. In the early stages of air filter analyses, it was also determined that the borosilicate filter should not have a polyester spun backing or be blended with an acrylic resin binder. Filter-strengthening additives increase sampling performance, yet they introduce additional analytical uncertainty by increasing the undigestible mass encountered during the acid digestion process. Currently, collection of perimeter air samples is performed at FMSS using the binder-less "Filter Paper Air Environmental (FPAE)" grade.

It should be noted that low concentrations of naturally-occurring radioactive materials (NORM) in borosilicate fiber media should be expected and must be accounted for in both the gross and isotopic analysis processes. Incorporating blank counting and radiochemistry blank analysis is critical when attempting to discern values near the DEL in the presence of background NORM in both the filter media and non-impacted air environment. To avoid batch-specific variances, blanks should be from the same production lot as the media used for sample collection.

### **Digestion**

Initial problems with digestion of glass fiber filters were encountered in September of 2004. An insoluble residue remained after the following steps were taken:

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ**  
**Abstract #8238**

- Cut the filter into predetermined size squares or rectangles using gloves. Make sure the filter has been folded so that the gloves only touch the clean side.
- The filter pieces are placed into glass beakers, which are arranged in a muffle furnace in a pattern that matches their placement in the muffle furnace with their sample identification number. This is done because any markings on the beaker shall be vaporized by the high furnace temperatures. Each beaker has the appropriate activity of tracer added and analyte spike for the LCS sample. The samples are gradually heated to 600°C. The temperature is held at 600°C for four hours, then the muffle furnace is turned off and the beakers allowed to cool.
- The filters are transferred from the glass beaker to a Teflon® beaker. The Teflon® beaker was first cleaned with 4M HNO<sub>3</sub> prior to adding the sample. After transferring the filters to the Teflon® beaker, 10 mls of concentrated HCl was added to the glass beaker, heated for 15-20 minutes, then poured into the Teflon® beaker.
- Rinse the glass beaker with 10 ml concentrated nitric acid and transfer to the Teflon® beaker. Add more concentrated HNO<sub>3</sub> and HCl in equal volumes so that the acid just covers the filters. Add 10 ml HF to the Teflon® beaker carefully.
- Digest on the hot plate for 4 hours. Start the temperature at 250C and increase the temperature as the volume of acid decreases. Evaporate to dryness.
- Add 10 ml conc. HCL, 10 ml conc. nitric, and 10 ml HF and redigest for another 4 hours, evaporating to dryness.

The problem was attributed to a binder in the filter. However, subsequent digestions with binderless filters also yielded an insoluble mass. The problem with an insoluble residue is that the analytes of concern may be irreversibly absorbed onto the insoluble material.

Communication with other laboratories indicated that a similar problem had been observed for an extended period of time. However, some labs would spike filter digests AFTER aliquots had been taken for the various analyses (leaving the insoluble material behind) so that they obtained good tracer recoveries. Thus, because of their procedure, their analyte results, especially for thorium, were biased low (possibly by a substantial amount). Gross counting of the undigested mass at the UFML identified the presence of bound beta-emitting radionuclides in the insoluble digestate above background. Given the nature of the insoluble mass and the limitations of gross counting, quantification of the activity or confirmation of the presence/absence of alpha activity was not possible.

UFML solicited advice from experts in the field of radiochemistry analysis, with three different experts actually visiting the Maywood site to assist UFML with their attempts to successfully digest glass fiber filters. Despite their best efforts, the UFML continued to obtain low and/or inconsistent results

The best results were obtained for blank filters processed and tested in November 2005. The UFML lab conducted a series of tests varying the number of times that the sample was heated in the muffle furnace, digested, and/or filtered (see the bulleted steps above). When filters were heated in a muffle furnace and digested two times each, very good tracer recoveries were obtained for U-232, and good to average recoveries were obtained for Th-232. The average and standard deviation for the U-232 recovery is  $93 \pm 9.1\%$  while the average Th-229 recovery is 70

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ**  
**Abstract #8238**

± 22% (four trials each). These tests also checked the amount of tracer that may have been left behind in the insoluble portion by performing a final muffle and digestion on the insoluble portion. The results indicate that average percentages of U-232 and Th-229 measured from the third extraction, were 0% and 3%, respectively. While these results were encouraging, two of the four filters recovered only 53% and 58% Th-229 from the three extractions. The percentage of Th-229 activity recovered from the other two filters was 91% and 92%. . While the low (53 and 58%) percentages are not terrible, the variability in the percent recoveries gives one less confidence in the data and forces the data user to be more conservative in their decision-making. The results of these tests are provided in Table I below.

Table I. Results Obtained for Th-229 and U-232 Tracers Following Multiple Muffle Furnace Heating and Digestion of Borosilicate Air Filters

Sample ID	Tracer Added	Tracer Activity, pCi	2σ un-certainty, pCi	MDA, pCi	Percent Recovery
4A	5.02 pCi of Th-229	4.48	0.41	0.04	89
4B		0.11	0.06	0.97	2
5A		2.66	0.31	0.04	53
5B		0.27	0.09	0.08	5
6A		4.54	0.41	0.02	90
6B		0.10	0.06	0.04	2
7A		2.51	0.29	0.10	50
7B		0.17	0.08	0.05	3
4A	4.61 pCi of U-232	4.64	0.43	0.02	101
4B		0.01	0.02	0.04	0
5A		4.10	0.40	0.05	89
5B		0.00	0.02	0.02	0
6A		4.61	0.41	0.04	100
6B		0.00	0.02	0.05	0
7A		3.78	0.37	0.03	82
7B		0.00	0.02	0.05	0

Table I Notes: For the A samples, perform the following on a blank filter with added tracer: muffle, digest, centrifuge, filter and rinse 3 times; set aside the solution and repeat the same steps on the insoluble portion. Combine the solutions, perform uranium and thorium separation using a resin column and count using alpha spectroscopy.

For the B samples, take the remaining insoluble material left after the first two rounds of muffling and digestion, muffle, digest, centrifuge, filter and rinse 3 times; take the solution, perform uranium and thorium separation using a resin column and count using alpha spectroscopy

In January 2006, method blanks and laboratory control samples (LCS) were analyzed using Hi-Q borosilicate filters. Method blanks are clean filters spiked with tracers prior to muffling, while LCS are clean filters spiked with tracers and the analytes of interest prior to muffling. Excellent recoveries were obtained for the uranium tracer and the U-234 and U-238 analytes. A measurable U-235 recovery was only obtained for LCS Run 2. For thorium, the results were variable. All target analyte recoveries are normalized based upon their associated tracer recovery. The results are provided in the Table II below.

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ  
Abstract #8238**

Table II. Iso-Uranium and Iso-Thorium Tracer and Blank Spike Recoveries from Borosilicate Glass Fiber Air Filters

Sample QC Type	Dates of Analysis	U-232 Tracer Percent Recovery	U-Natural Isotope Percent Recoveries
Method Blank	1/13/2006	111%	NA
LCS	1/13/2006	101% (Run 1) 104% (Run 2) 94.7% (Run 3)	U-234: 98.9, 94.5, 108%; U-235: 87.5% U-238: 94.9, 97.6, 111%
		Th-229 Tracer Percent Recovery	Th-230 Percent Recovery
Method Blank	1/13/2006	42.9%	NA
LCS	1/13/2006	98.5% (Run 1) 5.8% (Run 2) 14.4% (Run 3)	104% (Run 1) 182% (Run 2) 116% (Run 3)

### **Leaching**

UFML attempted to process air filters using a leach method in March 2006 on six batches using concentrated HNO<sub>3</sub> only or HNO<sub>3</sub> and HCl. It turns out that the filters were not actually leached but instead were simply digested with an acid mixture that did not include HF. The test batches varied specific steps within the digestion process. The results are provided in Table III below. Most of the tracer results are fairly low and further development is warranted before leaching can be considered a viable method. The Th-229 tracer results generally ranged from 25% to 45%. However, there are three results in Test #1 which yielded excellent recoveries between 77% and 87%. The U-232 tracer recoveries generally ranged from 35% to 60% with somewhat better results for Test #1. Also, the Test #2 U-232 tracer results suggest that no tracer was added. The Th-230 and U-238 LCS spike recoveries are very good. This is to be expected since the analyte results are normalized to the tracer recovery to obtain the analyte recovery.

### **SURROGATE EVALUATIONS UTILIZED**

Until a reliable method for analysis of filter media is identified and implemented the FMSS will continue the process of gross counting comparison with a DEL derived via surrogate analysis. A gross alpha counting DEL is calculated by evaluation of the uranium and thorium decay series ratios in excavation soil samples. The radionuclide blends in excavated soils are the best representation available of the radionuclide blends in air emissions.

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ  
Abstract #8238**

Table III. Filter "Leach Test" Results; Digestion with HCl and/or HNO<sub>3</sub> (HF was not used)

	<b>Th-229 Tracer Percent Recovery</b>	<b>Th-230 Spike Percent Recovery</b>	<b>U-232 Tracer Percent Recovery</b>	<b>U-238 Spike Percent Recovery</b>
<b>Test #1</b>				
Method Blank 1	87	---	61	---
Method Blank 2	79	---	60	---
LCS #1	77	89	90	70
LCS #2	44	107	66	75
<b>Test #2</b>				
Method Blank 1	35	---	1	---
Method Blank 2	34	---	2	---
LCS #1	42	96	1	87
LCS #2	42	94	1	77
<b>Test #3</b>				
Method Blank 1	36	---	30	---
Method Blank 2	34	---	40	---
LCS #1	37	99	39	97
LCS #2	40	91	35	104
<b>Test #4</b>				
Method Blank	24	---	41	---
LCS	27	97	53	85
REG	30	96	38	93
LREP	30	88	66	111
<b>Test #5</b>				
Method Blank	32	---	51	---
LCS	32	91	47	108
REG	42	94	43	104
LREP	50	90	47	109
<b>Test #6</b>				
Method Blank	41	---	51	---
LCS	43	86	61	81
REG	44	92	43	118
LREP	43	113	49	105

Table III Notes: For Test #1, new ¼ sections of borosilicate glass fiber filters were first heated in a muffle furnace for four hours, then acid leached with 15 ml 8M HNO<sub>3</sub> and 15 ml 6M HCl.

Test #2 is the same as Test #1 plus add watch glass while boiling acids with filter; Test #3 is the same as test #2 except no HCl was used during the digestion; Test #4 was the same as Test #3 except instead of digesting duplicate Method Blanks and LCS, one regular and one replicate sample were added which each consisted of approximately one gram of soil and 1/4 new filter; Test #5 was the same as Test #4 except samples were boiled to dryness instead of boiled down to approximately. 5 mLs prior to the resin separation process; Test #6 is the same as Test #5, however the remaining insoluble filter material were acid leached a second time.

**STRATEGIES for OVERCOMING RADIOCHEMISTRY METHOD DEFICIENCIES**

**Procedures Utilized by Offsite Laboratory –Digestion with Polyethylene Glycol Sequestration**



**WM'08 Conference, February 24-28, 2008, Phoenix, AZ**  
**Abstract #8238**

In September 2006, air filters were sent to a commercial laboratory for analysis. Similar to UFML, the commercial laboratory used an acid digestion procedure, however they added polyethylene glycol to “sequester” the silica, a process similar to complexation. Sequestration using polyethylene glycol appears to enhance the solubility of the actinides. The estimated MDAs for these methods are as follows:

Uranium 0.5 pCi/sample (Isotopic Uranium by Alpha Spectroscopy)  
 Thorium 1.0 pCi/ sample (Isotopic Thorium by Alpha Spectroscopy )  
 Ra-226 2 pCi/ sample (Method 903.1 Radon Emanation)  
 Ra-228 3 pCi/ sample (Method 9320 )

The commercial lab results are provided in Table IV.

Table IV. Results obtained by Commercial Laboratory from Analysis of Maywood Filters using Digestion with Polyethylene Glycol Sequestration

Sample	Analyte	Activity $\pm 2\sigma$ uncertainty or weight of barium for radium methods	Tracer Percent Recovery
#1	Th-228	0.234 $\pm$ 0.085	---
	Th-230	5.23 $\pm$ 0.84	---
	Th-232	0.218 $\pm$ 0.059	---
	Th-229 (4.503 pCi added)	2.73	60.7
#2	Th-228	1.62 $\pm$ 0.51	---
	Th-230	0.99 $\pm$ 0.36	---
	Th-232	1.14 $\pm$ 0.30	---
	Th-229 (22.51 pCi added)	13.7	60.6
#3	Th-228	2.78 $\pm$ 0.75	---
	Th-230	1.00 $\pm$ 0.40	---
	Th-232	1.28 $\pm$ 0.35	---
	Th-229 (22.51 pCi added)	11.9	52.7
#1	U-234	6.8 $\pm$ 1.1	---
	U-235	0.344 $\pm$ 0.085	---
	U-238	6.7 $\pm$ 1.1	---
	U-232 (4.475 pCi added)	3.25	72.5
#2	U-234	1.30 $\pm$ 0.33	---
	U-235	0.107 $\pm$ 0.099	---
	U-238	1.26 $\pm$ 0.30	---
	U-232 (22.38 pCi added)	17.1	76.5
#3	U-234	1.74 $\pm$ 0.39	---
	U-235	0.074 $\pm$ 0.071	---
	U-238	1.58 $\pm$ 0.37	---

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ  
Abstract #8238**

	U-232 (22.38 pCi added)	15.9	71.2
#1	Ra-226	23.8 ± 4.6	
	Barium (44.2 mg added)	45.4 mg	103
#2	Ra-226	1.12 ± 0.60	
	Barium (38.5 mg added)	39.3 mg	102
#3	Ra-226	2.09 ± 0.65	
	Barium (35.9 mg added)	39.7 mg	111
#1	Ra-228	21.7 ± 7.5	
	Barium (61.8 mg added)	50.4 mg	81.5
#2	Ra-228	4.0 ± 3.4	
	Barium (54.6 mg added)	57.7 mg	106
#3	Ra-228	4.0 ± 4.0	
	Barium (54.6 mg added)	48.5 mg	88.8

The tracer recoveries are very consistent for all methods. The relative standard deviation of the tracer percent recoveries were 7.9%, 3.8%, 4.7%, and 13.7% for Th-229, U-232, barium (Ra-226), and barium (Ra-228), respectively. In addition, the batch QC results were all within QC acceptance criteria.

For every analyte, sample #1 is a performance evaluation (PE) air filter sample. The PE results comparison is shown in Table V.

Table V. Commercial Laboratory Results for Performance Evaluation (PE) Air Filter Sample

Analyte	Commercial Laboratory Result, pCi	Activity of Spike, pCi	Percent Recovery
U-234	6.80	6.745	100.8
U-235	0.34	0.317	108.5
U-238	6.70	6.887	97.3
Th-230	5.23	5.054	103.5
Ra-226	4.76	5.014	94.9
Ra-228	4.34	6.745	64.3

With the exception of Ra-228, the recovery of which is a bit low, all of the PE sample results compare very favorably to the true values. The commercial laboratory methods using acid digestion followed by sequestration using polyethylene glycol appears to be a good method for accurate quantitation of the activities of these radionuclides on borosilicate air filters.

**Procedures Utilized by Offsite Laboratories –Potassium Pyrosulfate Fusion**

Fusion is a method where the sample is melted with a salt such as sodium carbonate, potassium hydroxide, etc. at high temperatures to form a fused melt. The melt is then cooled and the solid mass is dissolved in dilute acid.

Air filter samples were sent to a commercial laboratory and were analyzed this time by potassium pyrosulfate fusion for isotopic thorium, isotopic uranium, and Ra-226. A fusion cake is first produced with potassium fluoride which is effective in dissolving silica, refractory silicates and some metallic oxides. The results of these analyses are provided in Table VI.

Table VI. Isotopic Analysis Results for Borosilicate Air Filters Prepared by Pyrosulfate Fusion

Sample	Tracer or Carrier	Spike Amount (pCi, except for barium in µg)	Result (pCi, except for barium in µg)	Percent Recovery
S1	Barium	14920	18480	124
S1 dupl		14200	18320	129
S2		16050	19780	123
S3		13570	14950	110
S1	Th-229	2.24	1.65	74
S1 dupl		2.24	1.50	67
S2		2.24	1.67	74
S3		2.24	1.52	68
S1	U-232	4.48	3.14	70
S2		4.48	3.83	86
S3		4.48	3.82	85

The tracer percentage recoveries range from 67-74% for Th-229, 70-85% for U-232, and 110-129% for barium (Ra-226). The barium carrier recoveries are considered high as default limits for this recovery are typically 40-110%. The main drawback to the fusion is the cost, since platinum crucibles are the preferred crucible material, and they are quite expensive.

**CONCLUSIONS**

The UFML had limited success analyzing air filters for isotopic thorium and isotopic uranium after numerous attempts using a variety of sample preparation techniques as well as soliciting the advice of industry experts. The best results appeared to be obtained when preparation steps (heating in a muffle furnace, digestion with an acid mixture, centrifuging and filtering) were performed a second time on the insoluble material obtained from the first preparation sequence. Leaching, or digestion of the glass fiber without the use of HF, may hold promise but requires more development since the tracer recoveries were somewhat low. Employing an outside laboratory, we found two preparation methods, digestion using polyethylene glycol sequestration, and potassium pyrosulfate fusion, for which results were acceptable to very good. Both sequestration and fusion do a better job at getting the analytes of concern into the dissolved phase with less effort. Fusion has the drawback of being expensive due to the high cost of

**WM'08 Conference, February 24-28, 2008, Phoenix, AZ**

**Abstract #8238**

platinum crucibles, and so may not be the method of choice for laboratories that analyze a relatively small number of samples. Future enhancements such as grinding the air filters to a fine powder before digestion, and use of HF only during the initial digestion phase may lead to more accurate and reproducible results.