

November 10, 2016

Mr. Bradley Vann Remedial Project Manager U.S. Environmental Protection Agency, Region 7 11201 Renner Boulevard Lenexa, Kansas 66219

Subject:Radon Emanation Coefficient Study<br/>West Lake Landfill Site, Bridgeton, Missouri<br/>CERCLIS ID: MOD079900932<br/>EPA Region 7, START 4, Contract No. EP-S7-13-06, Task Order No. 0007<br/>Task Monitor: Bradley Vann, Remedial Project Manager

Dear Mr. Vann:

Tetra Tech, Inc. is submitting the attached report regarding a study evaluating radon emanation coefficient of core samples collected at the West Lake Landfill Site (WLLS) in Bridgeton, Missouri. If you have any questions or comments, please contact me at (816) 412-1775.

Sincerely,

Robert Monnig, PE

START Project Manager

Ted Faile, PG, CHMM START Program Manager

Enclosures

cc: Debra Dorsey, START Project Officer (cover letter only)

## **RADON EMANATION COEFFICIENT STUDY**

## WEST LAKE LANDFILL SITE BRIDGETON, MISSOURI CERCLA ID: MOD079900932

## Superfund Technical Assessment and Response Team (START) 4 Contract No. EP-S7-13-06, Task Order No. 0007

Prepared For:

U.S. Environmental Protection Agency Region 7 Superfund Division 11201 Renner Blvd. Lenexa, Kansas 66219

November 10, 2016

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#### **EXECUTIVE SUMMARY**

The Tetra Tech, Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) was tasked by the U.S. Environmental Protection Agency (EPA) to assist with a study to evaluate the radon emanation characteristics of core samples collected at the West Lake Landfill site (WLLS) in Bridgeton, Missouri. The purpose of this study was to determine whether actions of a sub-surface smoldering event (SSE) could cause physical or chemical changes to the radiologically impacted material (RIM) so as to alter the material's radon emanation coefficient (REC). The REC quantifies the fraction of radon that escapes from solid material into the adjacent pore space, and is a measurable parameter of particular interest for modeling radon release.

For the study, EPA obtained samples of materials from various locations of Operable Unit 1 (OU1) Areas 1 and 2 that had exhibited elevated gamma activity—an indication that the material contained RIM. The samples were subjected to various thermal and moisture adjusting conditions, and then underwent assessment for changes in the amount of radon emanating from the treated samples. Plots of REC results against moisture content revealed that REC generally increases with increased moisture content, a relationship that has also been demonstrated in previous studies. Influence of thermal treatment was also investigated by comparing REC results from tests under conditions of similar moisture content but differing thermal treatments. Overall, this comparison revealed no obvious tendency for thermal treatment alone to result in higher or lower RECs; thus, it does not appear that thermal treatment of a RIM-containing sample alters the REC.

#### 1.0 INTRODUCTION

The Tetra Tech, Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) was tasked by the U.S. Environmental Protection Agency (EPA) to assist with a study to evaluate the radon emanation characteristics of core samples collected at the West Lake Landfill site (WLLS) in Bridgeton, Missouri. START's tasks included: (1) engaging an analytical laboratory capable of preparing and implementing an analytical procedure to determine the radon emanation coefficient of core samples, (2) assisting with reception of core samples from the responsible parties (RP) and coordinating shipment of samples to the laboratory, (3) assisting EPA with data acquisition and management, and (4) documenting the study efforts.

The purpose of this study was to determine whether actions of a sub-surface smoldering event (SSE) could cause physical or chemical changes to the radiologically impacted material (RIM) so as to alter the material's radon emanation coefficient (REC). The REC quantifies the fraction of radon that escapes from solid material into the adjacent pore space, and is a measurable parameter of particular interest for modeling radon release. For the study, EPA obtained samples of materials from various locations of Operable Unit 1 (OU1) Areas 1 and 2 that had exhibited elevated gamma activity—an indication that the material contained RIM. Five of the six samples were obtained from previously collected subsurface core samples that had been preserved by the RPs. The remaining sample was collected by EPA from the surface of OU1 Area 2. The samples were subjected to various thermal and moisture adjusting conditions, and then underwent assessment for changes in the amount of radon emanating from the treated samples. These data will be used to inform investigators about potential effects of SSE on radon emanation rates of RIM within WLLS.

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### 2.0 PROBLEM DEFINITION, BACKGROUND, AND SITE DESCRIPTION

West Lake Landfill is an approximately 200-acre property that includes (1) several closed solid waste landfill units which accepted wastes for landfilling from the 1940s or 1950s through 2004, (2) a solid waste transfer station, (3) a concrete plant, and (4) an asphalt batch plant. The WLLS is at 13570 St. Charles Rock Road in Bridgeton, St. Louis County, Missouri, approximately 1 mile north of the intersection of Interstate 70 and Interstate 270 (see Appendix A, Figure 1). The WLLS was used for limestone quarrying and crushing operations from 1939 through 1988. Beginning in the late 1940s or early 1950s, portions of the quarried areas and adjacent areas were used for landfilling municipal refuse, industrial solid wastes, and construction/demolition debris. In 1973, approximately 8,700 tons of leached barium sulfate residues (a remnant from the Manhattan Engineer District/Atomic Energy Commission project) were reportedly mixed with approximately 39,000 tons of soil from the 9200 Latty Avenue site in Hazelwood, Missouri, transported to the WLLS, and used as daily or intermediate cover material. In December 2004, the Bridgeton Sanitary Landfill—the last landfill unit to receive solid waste—stopped receiving waste pursuant to an agreement with the City of St. Louis to reduce potential for birds to interfere with Lambert Field International Airport operations. In December 2010, Bridgeton Landfill detected changes-elevated temperatures and elevated carbon monoxide levels-in its landfill gas extraction system in use at the South Quarry of the Bridgeton Sanitary Landfill portion of the site (a landfill portion not associated with known RIM). Further investigation indicated that the South Quarry Pit landfill was undergoing an exothermic SSE. EPA conducted this radon emanation study to inform investigators about potential effects of an SSE on radon emanation rates.

## 3.0 SAMPLING PROCESS DESIGN AND RATIONALE

The following design and rationale for the sampling process during this study was presented in the EPA-approved Quality Assurance Project Plan (QAPP) prepared for the study (Tetra Tech 2015). The sampling process design followed the EPA 7-step process of establishing data quality objectives (DQO).

## Step 1 – State the Problem

## Problem Statement

An SSE in one of the non-radiological disposal cells has been reported. Some have hypothesized that an SSE could increase the rate of radon release from the subsurface. A measurable parameter of particular interest for modeling radon release is the REC. Although literature values from previous studies of RECs of various materials (mostly uranium tailings and soils) are available, it was deemed desirable to obtain site-specific RECs determined experimentally via a bench-scale study using West Lake RIM. Moreover, the bench-scale study could determine whether actions of an SSE could cause physical or chemical changes to the RIM that alter the material's REC.

## Conceptual Site Model of Environmental Hazard to be Evaluated

An SSE event could cause a change in subsurface moisture and/or the physical/chemical makeup of the RIM, which may affect the REC. The REC quantifies the fraction of radon that escapes from solid material into the adjacent pore space (see Strong and Levins 1982). Experimentally determined RECs of RIM-containing material subjected to conditions of various moisture content and thermal treatment may inform investigators about effects of an SSE on radon emanation rates.

## Alternative Approaches

In the absence of experimentally determined RECs, investigators could evaluate sensitivity of modeled radon release rates to a range of RECs and other parameters related to subsurface conditions (such as moisture content). A review of published literature could inform investigators regarding the probable range of the REC.

## Step 2 – Identify the Decision

#### Principal Study Question

Experimental data will be used to answer the following principal study questions:

- Question 1: What is/are the REC(s) of RIM-containing samples at various moisture contents?
- Question 2: Does thermal treatment of a RIM-containing sample alter the sample's REC?

#### Decision Statement / Alternative Actions

No decision statement or alternative actions are associated with this study. The study will inform data users regarding radon emanation characteristics of RIM-containing samples subjected to moisture and thermal treatment.

## Step 3 – Identify Inputs to the Decision

The principal study questions will be answered by experimentally determining RECs of RIM-containing samples collected at OU1 of the West Lake Landfill. Measuring a REC involves placing a radium-226-impacted material within an air-tight chamber, allowing accumulation of radon gas within the sealed chamber for a period of time, and then determining radon concentration in the chamber air and radium-226 concentration in the impacted material.

#### Step 4 – Define the Boundaries of the Study

#### Target Population

The target population is the West Lake Landfill RIM from Areas 1 or 2 of OU1 (see Appendix A, Figure 1). Samples from Area 1 or 2 of OU1 that exhibit significantly elevated gamma activity will presumably contain RIM, and selected samples will be submitted for laboratory determination of RECs. Submitted samples will also undergo laboratory analysis for uranium/thorium isotopes and radium-226; data then can be used to evaluate if the sample is representative of radionuclide concentrations historically detected in RIM-containing samples.

#### Spatial and Temporal Boundaries

Samples will be collected from Area 1 or 2 of OU1 of the West Lake Landfill, and will be selected to represent RIM-containing material. Temporal boundaries are not a significant aspect of this study; the half-life of radium-226, the parent radionuclide of radon-222, is about 1,600 years.

## Practical Constraints on Acquiring the Data

No practical constraints have been identified.

## Define the Scale of Inference

RECs determined under conditions of various moisture content and thermal treatment would be representative of the site and could be used in future radon modeling efforts. The study would not provide information pertaining to changes in radon flux related to landfill settling, development of fissures in cover due to drying, or any advective transport of radon.

## Steps 5 and 6 - Develop a Decision Rule and Specify Tolerable Limits on Decision Errors

The study will provide data regarding RECs of RIM-containing materials at various levels of moisture content, and under pre- and post-thermal treatment conditions. Alternative actions related to this study have not been identified; therefore, a decision rule and specification of tolerable limits on decision errors are not needed.

## Step 7 – Optimize the Design for Obtaining Data

Previous studies have characterized RECs of soils, uranium ores, and uranium tailings. The Argonne National Laboratory (ANL) summarized several studies in *Data Collection Handbook to Support Modeling Impacts of Radioactive Material in Soil*; in these studies, the REC was found to vary from 0.02 to 0.70 in soils (ANL 1993).

Data from previous studies also demonstrate a strong influence of moisture content on RECs. This exhibit from the Strong and Levins (1982) study of uranium mill tailings illustrates the influence of moisture content determined in their study:



FIG. 2. Variation of emanation coefficient with moisture content for Jabiluka tailings.

Effects of heating on emanation coefficients have also been studied. Garver and Baskaran (2004) found that minerals in their study that had been heated to 600 degrees Celsius (°C) exhibited lower RECs than studied minerals that had not been heated. The researchers hypothesized that heating anneals nuclear tracks (created from previous decay events) within the mineral that serve as conduits for release of radon.

#### 4.0 SAMPLE COLLECTION

For the study, EPA obtained samples of materials from various locations of OU1 Areas 1 and 2 that had exhibited elevated gamma activity—an indication that the material contained RIM. Five of the six samples were obtained from previously collected subsurface core samples that had been preserved by the RPs. The remaining sample was collected by EPA from the surface of OU1 Area 2. Table 1 describes the samples collected and submitted to Southwest Research Institute (SwRI), the analytical laboratory that designed the test procedures and conducted the radon emanation study.

#### TABLE 1

Sample Name	Date Collected	Location Description	Sample Depth (ft bgs)
1D7-84-85	11/16/2015	Obtained from a previously collected and preserved soil core collected by the Responsible Parties via sonic drilling at location "1D7" in OU1 Area 2.	84 - 85
A1-AC-1	12/21/2015	Obtained from a previously collected and preserved soil core collected by the Responsible Parties via sonic drilling at location "A1-AC-1" in OU1 Area 1.	10 - 11
A1-AC-3	12/21/2015	Obtained from a previously collected and preserved soil core collected by the Responsible Parties via sonic drilling at location "A1-AC-3" in OU1 Area 1.	5-6
A2-AC-21	12/21/2015	Obtained from a previously collected and preserved soil core collected by the Responsible Parties via sonic drilling at location "A2-AC-21" in OU1 Area 2.	12 – 13
A2-S1	11/24/2015	Collected specifically for this study via use of a hand trowel from the surface of OU1 Area 2 within an area exhibiting elevated gamma activity.	0-0.5
WL-234CT	1/8/2016	Obtained from a previously collected and preserved soil core collected by the Responsible Parties via sonic drilling at location "WL-234CT" in OU1 Area 2.	8 - 10

## CORE SAMPLES FOR RADON EMANATION STUDY

Note:

ft bgs Feet below ground surface

### 5.0 ANALYTICAL PROCEDURE AND TEST CONDITIONS

As proposed in the QAPP, the samples for this study were subjected to multiple test conditions, each of which regarded a particular thermal treatment and target moisture content, and a REC was determined for each treatment of the sample. Thermal treatments included subjecting samples to temperatures of 105 °C and 250 °C for 16 or 48 hours. The 105 °C treatment temperature was selected to simulate loss of liquid water from the sample at temperatures near the boiling point of water. The 250 °C temperature was selected to potentially induce smoldering of the sample and to approximate the upper end of the expected temperature induced by an SSE in the landfill. Two durations of thermal treatment—16 and 48 hours— were selected to assess for possible variation in the REC due to varying degrees of drying/smoldering of the samples. In all, SwRI subjected the samples to nine unique test conditions described in Table 2. In addition to measuring parameters directly related to radon emanation, SwRI also analyzed the samples for other naturally occurring radionuclides (including uranium and thorium isotopes) and metals. Full analytical results and a detailed description of the radon emanation testing procedure are in SwRI's report conveyed separately in Tetra Tech 2016.

## TABLE 2

Test Condition	Thermal Treatment	Moisture Treatment	Objective of Test Condition
1	None (as received)	None. Samples maintained at their "as received" moisture content (14 to 27 wt % moisture content)	Measure the REC of RIM not subjected to heating and moisture altering conditions of an SSE.
2	Heated at 105 °C (221 °F) for 16 hours	As found after thermal treatment (near 0 wt % moisture content)	Measure the REC of RIM subjected to moderate heating and drying that buried waste may undergo near an SSE.
3	Heated at 250 °C (482 °F) for 16 hours	As found after thermal treatment (near 0 wt % moisture content)	Measure the REC of RIM subjected to more intense heating and drying that buried waste may undergo at or very near an SSE.
4		"As Found" after thermal treatment (near 0 wt % moisture content)	Evaluate whether extending the "direct SSE" heating period affects the REC.
5	Heated at 250 °C (482 °F) for 48 hours	Moisture content adjusted to approximate the "as received" moisture content (14 to 26 wt % moisture content)	Measure the REC of RIM subjected to heating treatment approximating an SSE, and whose moisture content has subsequently returned to "as received" moisture content.
6		Water added to bring moisture content of sample to approximately 10 wt %	Measure the REC of RIM subjected to heating treatment approximating an SSE, and whose moisture content subsequently becomes relatively high.
7	None (as received)	Water added to bring sample to a saturated moisture content (26 to 41 wt % moisture content)	
8	None (minimal heating at 60 °C [140 °F] to achieve	Water added, followed by drying until moisture content approximates 2 wt %	Evaluate effect of moisture content of RIM not subjected to heating of an SSE.
9	desired moisture content)	Water added, followed by drying until moisture content approximates 10 wt %	

# THERMAL AND MOISTURE TEST CONDITIONS

Notes:

°C °F

wt %

REC

Degrees Celsius Degrees Fahrenheit Percent by weight Radon emanation coefficient Radiologically impacted material Sub-surface smoldering event RIM

SSE

## 6.0 DATA VALIDATION, VERIFICATION, AND USABILITY

START reviewed and qualified the data according to EPA Contract Laboratory Program guidelines (EPA 2008), the *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (EPA 2004), and other criteria specified in the applicable methods. Findings of this review are documented in the data validation report in Appendix D. The following are brief descriptions of the suggested qualifiers:

- <u>Radon Emanation Analysis</u> Using additional sample aliquots collected from the homogenized sample, the laboratory performed one duplicate determination of the REC (via radon in-growth and radium-226 and radon-222 analyses) for each test condition. Relative percent differences (RPD) between equilibrium concentrations in the duplicate pairs were less than 25%, except for radon-222 equilibrium concentrations in sample 1D7-84-85/Condition 5 (analysis and re-analysis resulted in RPDs of 49.9% and 43.9%) and sample A2-S1/Condition 6 (analysis and re-analysis resulted in RPDs of 85.6% and 87.1%). These duplicate pair results of radon-222 equilibrium concentrations and the associated RECs (yielded, in part, by radon-222 results) are suggested to be qualified as estimated and flagged "J". Data users should be aware that similar irregularities may exist in concentrations in other samples that were subjected to the different conditions but did not undergo duplicate analysis.
- <u>Thorium-228 Analysis</u> Some thorium-228 results only slightly exceeded thorium-228 concentrations in the laboratory blanks; therefore, these results are suggested to be qualified as estimated, possibly biased high, and flagged "J".
- <u>Lanthanum Analysis</u> Lanthanum recoveries were 59 and 46 percent, below their quality control (QC) limits of 75 to 125 percent. The lanthanum results from all samples are suggested to be qualified as estimated, possibly biased low, and flagged "J".
- <u>Tin Analysis</u> Duplicate analyses for tin resulted in exceedance of the RPD QC limit of 20 percent, with an RPD of 30.5 percent, indicating a heterogeneous distribution of the metal. Due to this uncertainty, the result for tin in sample A2-S1 is suggested to be qualified as estimated and flagged "J".

Overall, review of the laboratory analytical report indicated that quality of the data was acceptable and usable as qualified for the intended purposes of those data.

## 7.0 CHARACTERISTICS OF THE RADON EMANATION SAMPLES

Various analyses, in addition to radon emanation testing, occurred to characterize moisture content and temperature stability (via thermogravimetric analysis), and to confirm presence of RIM (via radionuclide analysis). The following sections discuss these analyses.

## 7.1 Thermogravimetric Analysis

Each sample underwent a thermogravimetric analysis (TGA) that involved weighing an aliquot of the sample (as received, prior to any intentional heating or drying of the sample) and heating it at a specified rate for a specified period of time. During the heating period, the sample weight was monitored, and thermal detectors were used to determine occurrence of endothermic or exothermic reactions. The resulting weight versus time curve is useful for indicating reactions occurring within the sample. TGA of the radon emanation samples was intended to verify assumptions regarding anticipated water loss versus temperature, and to identify occurrence of combustion of the sample at elevated temperatures. These TGA analyses are presented in SwRI's analytical report (see Tetra Tech 2016). Overall, the only effect obvious from the TGA results at temperatures up to 250 °C (a temperature greater than the approximate upper end of expected temperature induced by an SSE in the landfill) was evaporation of water from the sample. Each sample lost approximately 15 to 25 percent of its weight, apparently from water loss, upon reaching a temperature of about 105 °C. Beyond 105 °C, the rate of sample weight loss (presumably from additional water loss) with increasing temperature decreased substantially. Based on TGA results, thermal treatment conditions were left unchanged.

#### 7.2 Uranium, Thorium, and Radium Content

Each sample underwent analyses for uranium, thorium, and radium isotopes—data useful for evaluating if the sample was representative of radionuclide concentrations historically detected in RIM-containing samples. Results of these analyses appear in Appendix B, Table B-1. Each sample exhibited elevated concentrations of natural uranium, thorium-230, and radium-226, indicating presence of RIM. Radium-226 results ranged from 196 to 3,430 pCi/g, thorium-230 results ranged from 6,880 to 58,800 pCi/g, and uranium-238 results ranged from 73.1 to 816 pCi/g. These ranges exceeded established reference levels—7.9 pCi/g combined radium, 7.9 pCi/g combined thorium, and 54.5 pCi/g total uranium (Engineering Management Support, Inc. 2011)—thus indicating that the samples were characteristic of RIM.

## 7.3 Abundance of Uranium Isotopes

Each sample was analyzed for uranium isotopes (uranium-234, -235, -236, and -238) via a method utilizing inductively coupled plasma mass spectrometry (ICP-MS). This sensitive analysis yields isotopic uranium and total uranium concentrations reported on a mass basis (e.g., milligrams per kilogram) (see Appendix B, Table B-2). Isotopic uranium results revealed relative abundance of isotopes within each sample, and allowed a comparison of isotopic composition to that of natural uranium—that is, uranium containing relative concentrations of isotopes found in nature (approximately 0.7 percent uranium-235, 99.3 percent uranium-238, and trace amounts of uranium-234 and uranium-236) (Nuclear Regulatory Commission [NRC] 2016, and Steier et al. 2008). This comparison, presented in Table B-2 of Appendix B, showed that the uranium isotopic composition of each sample strongly corresponded to the isotopic composition of natural uranium, indicating that uranium within the samples was characteristic of natural uranium.

## 8.0 EVALUATION OF RADON EMANATION STUDY RESULTS

The following sections present evaluations of the REC results with respect to the various moisture and thermal treatments. Tabulated REC data are in Appendix B, Table B-3 and detailed individual sample plots of RECs determined under conditions of various moisture contents and thermal treatments are in Appendix C. The laboratory analytical report from SwRI is conveyed separately in Tetra Tech 2016.

## 8.1 VARIATION OF REC WITH MOISTURE CONTENT

Because data from previous studies have demonstrated a strong influence of moisture content on RECs, REC results were first plotted against moisture content to evaluate if this affect was evident. Exhibit 1 shows this plot of moisture content vs REC for each of the six samples. Examination of these plots confirms the anticipated influence of moisture content on RECs—that is, the REC generally increases with increased moisture content. This relationship is particularly evident on plots of samples A1-AC-1 and A1-AC-3.

## **EXHIBIT 1**



## VARIATION OF RADON EMANATION COEFFICIENT WITH MOISTURE CONTENT AND THERMAL TREATMENT

X9025.14.0007.000

## 8.2 VARIATION OF REC WITH THERMAL TREATMENT

The various thermal treatments are represented by color of data points plotted on Exhibit 1. Review of Exhibit 1 reveals no strong relationship between thermal treatment and REC.

The influence of thermal treatment is further investigated in Exhibits 2, 3, and 4, which compare REC results among test conditions involving similar moisture content but differing thermal treatments. Exhibit 2 compares results among treatments with near 0 percent moisture content, Exhibit 3 compares treatments with moisture contents near 10 percent, and Exhibit 4 compares treatments with "as received" moisture contents (ranging from 14 to 27 percent). The exhibits present results by sample, ranked from highest to lowest REC, and color coded to indicate the various thermal treatments. If thermal treatment strongly influenced REC (given similar moisture content), a pronounced pattern should be apparent in the exhibits (e.g., if increased thermal treatment were to cause increased REC, the 250 °C/48 hour treatment results—those colored red—should dominate the top row of each of the exhibits; or conversely, the bottom row if increased thermal treatment resulted in lower RECs).

## **EXHIBIT 2**

	1D7-84-85	A1-AC-1	A1-AC-3	A2-AC-21	A2-S1	WL-234CT
Highest REC	Condition 2 REC: 0.0514 MC: 0.28	Condition 3 REC: 0.0452 MC: 0.0	Condition 2 REC: 0.0525 MC: 0.2	Condition 2 REC: 0.228 MC: 0.0	Condition 2 REC: 0.0519 MC: 0.14	Condition 2 REC: 0.0547 MC: 0.32
Hi	Condition 3 Dup REC: 0.042 MC: 0.0	Condition 2 REC: 0.0383 MC: 0.02	Condition 3 REC: 0.0459 MC: 0.0	Condition 4 REC: 0.13 MC: 0.0	Condition 2 Dup REC: 0.0517 MC: 0.09	Condition 3 REC: 0.0435 MC: 0.0
	Condition 3 REC: 0.0411 MC: 0.0	Condition 4 REC: 0.033 MC: 0.0	Condition 4 REC: 0.0435 MC: 0.0	Condition 3 REC: 0.124 MC: 0.0	Condition 4 Dup REC: 0.0499 MC: 0.0	Condition 4 REC: 0.0287 MC: 0.0
t REC	Condition 4 REC: 0.0366 MC: 0.0				Condition 4 REC: 0.044 MC: 0.0	
Lowest REC					Condition 3 REC: 0.0306 MC: 0.0	
	Notes			T	nermal Treatment Ke	
				105 °C / 16 hrs	250 °C / 16 hrs	250 °C / 48 hrs
	Dup	Laboratory duplica			250 C / 10 ms	250 C / 48 1115
	MC	Moisture content,	percent by weight			
	REC	Radon emanation				

## VARIATION OF RADON EMANATION COEFFICIENT WITH THERMAL TREATMENT AT LOW MOISTURE CONDITIONS

Exhibit 2 possibly shows a tendency for Condition 2 treated samples to exhibit higher RECs; however and notably, the Condition 2 treated samples generally contained more moisture, likely causing increased REC. The greater moisture in the Condition 2 treated samples is expected, as Condition 2, 3, and 4 treated samples were measured following thermal treatment and without additional moisture adjustment, and Condition 2 treated samples received the least amount of thermal treatment. Comparing Conditions 3 and 4 reveals no obvious tendency for increased thermal treatment (16 vs 48 hours) to result in higher or lower RECs.

## EXHIBIT 3

## VARIATION OF RADON EMANATION COEFFICIENT WITH THERMAL TREATMENT AT 10 PERCENT MOISTURE CONTENT

U	1D7-84-85	A1-AC-1	A1-AC-3	A2-AC-21	A2-S1	WL-234CT
Highest REC	Condition 6 REC: 0.134 MC: 9.79	Condition 6 REC: 0.12 MC: 10.27	Condition 9 REC: 0.143 MC: 6.62	Condition 9 REC: 0.391 MC: 10.66	Condition 6 REC: 0.139 J MC: 10.47	Condition 9 REC: 0.107 MC: 12.73
	Condition 9 REC: 0.0541 MC: 8.19	Condition 9 REC: 0.0989 MC: 6.7	Condition 6 REC: 0.139 MC: 10.44	Condition 6 REC: 0.336 MC: 10.12	Condition 9 Dup REC: 0.139 MC: 8.78	Condition 6 REC: 0.0894 MC: 10.08
REC					Condition 9 REC: 0.121 MC: 8.53	
Lowest REC					Condition 6 Dup REC: 0.0619 J MC: 9.94	
	Notes				<b>The sum of Tree</b>	too ant Mari
		Loboratory duplica	*~		Thermal Trea None (< 60 °C)	250 °C / 48 hrs
	Dup	Laboratory duplica			Tied Ranking	250 07 40 1113
	MC	Moisture content,			neo kanking	
	REC	Radon emanation	coefficient			

Comparing Condition 6 (heated to 250 °C for 48 hours and then adjusted to a 10 percent moisture content) and Condition 9 (no thermal treatment and adjusted to a 10% moisture content) in Exhibit 3, no tendency is obvious for thermal treatment to result in higher or lower RECs.

## EXHIBIT 4

## VARIATION OF RADON EMANATION COEFFICIENT WITH THERMAL TREATMENT AT "AS RECEIVED" MOISTURE CONTENT

U	1D7-84-85	A1-AC-1	A1-AC-3	A2-AC-21	A2-S1	WL-234CT
Highest REC	Condition 5 Dup REC: 0.171 J MC: 20.32	Condition 5 REC: 0.131 MC: 14.86	Condition 5 REC: 0.164 MC: 13.86	Condition 1 REC: 0.473 MC: 18.65	Condition 5 REC: 0.141 MC: 23.79	Condition 1 REC: 0.136 MC: 26.63
^	Condition 1 REC: 0.14 MC: 18.94	Condition 1 REC: 0.128 MC: 14.95	Condition 1 REC: 0.14 MC: 13.67	Condition 5 REC: 0.376 MC: 18.64	Condition 1 REC: 0.0664 MC: 21.24	Condition 5 REC: 0.0907 MC: 26
3EC	Condition 1 Dup REC: 0.138 MC: 18.94					
Lowest REC	Condition 5 REC: 0.0898 J MC: 19.06					
	Notes				Thermal Treatment	
	Dup	Laboratory duplica	te		None (< 60 °C)	250 °C / 48 hrs
	MC	Moisture content,	percent by weight			
	REC	Radon emanation	coefficient			

Comparing Condition 5 (heated to 250 °C for 48 hours and then adjusted to the sample's "as received" moisture content) and Condition 9 (no thermal treatment and at the sample's "as received" moisture content) in Exhibit 4, no tendency is obvious for thermal treatment to result in higher or lower RECs.

Overall, comparison of various thermal treatments among samples with similar moisture content reveals no obvious tendency for thermal treatment to result in higher or lower RECs.

# 8.3 EVALUATION OF THE STUDY RESULTS WITH RESPECT TO THE PRINCIPAL STUDY QUESTIONS

The QAPP presented these study questions:

- Question 1: What is/are the REC(s) of RIM-containing samples at various moisture contents?
- Question 2: Does thermal treatment of a RIM-containing sample alter the sample's REC?

Study Question 1 can be answered by reviewing the REC results with respect to the various moisture contents. Appendix B lists the REC results with respect to the various moisture content test conditions.

In addition, the data show a strong influence of moisture content on RECs, as had been demonstrated in previous studies.

Regarding Question 2, as discussed in Section 8.2, the data do not appear to reveal a tendency for thermal treatment alone to alter RECs of RIM-containing samples.

## 9.0 SUMMARY OF OBSERVATIONS

A bench-scale study using West lake RIM has been conducted to determine whether actions of an SSE could cause physical or chemical changes to the RIM that may alter the material's REC. A REC quantifies fraction of radon that escapes from solid material into the adjacent pore space, and is a measurable parameter of particular interest for modeling radon release. For the study, EPA obtained samples of materials from various locations of OU1 Areas 1 and 2 that had exhibited elevated gamma activity—an indication that the materials contained RIM. Five of the six samples were obtained from previously collected subsurface core samples that had been preserved by the RPs. The remaining sample was collected by EPA from the surface of OU1 Area 2.

The samples were subjected to multiple test conditions, each of which regarded a particular thermal treatment and target moisture content, and a REC was determined for each treatment of each sample. The thermal treatments included subjecting samples to temperatures of 105 °C and 250 °C. The 105 °C treatment temperature was selected to simulate loss of liquid water from the sample at temperatures near the boiling point of water. The 250 °C temperature was selected to induce smoldering of the sample and to approximate the upper end of the expected temperature range induced by an SSE in the landfill. Two durations of thermal treatment—16 and 48 hours—were selected to assess for possible variation in the REC due to varying degrees of drying/smoldering of the samples.

The influence of moisture content was first investigated because a literature review had revealed previous studies demonstrating a strong influence of moisture content on RECs. To investigate this relationship, REC results were plotted against moisture content to assess if that previously detected effect would be readily apparent. Plots of moisture content vs REC for each of the six samples indeed confirmed the anticipated influence of moisture content on the RECs—that is, the REC generally increases with increased moisture content.

Influence of thermal treatment was also investigated by comparing REC results from tests under conditions of similar moisture content but differing thermal treatments. The data were first categorized by moisture content—near 0 percent moisture content, 10 percent moisture content, and "as received" moisture content (ranging from 14 to 27 percent); then for each sample, the various thermal treatments were ranked according to the measured REC. Overall, this comparison revealed no obvious tendency for thermal treatment alone to result in higher or lower RECs; thus, based on temperatures and durations studied (up to 250 °C for 48 hours), it does not appear that thermal treatment of a RIM-containing sample alters the REC.

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APPENDIX A

FIGURE



**APPENDIX B** 

TABULATED RESULTS OF RADON EMANATION STUDY

#### TABLE B-1

## URANIUM, THORIUM, AND RADIUM CONTENT OF SAMPLES

	Radionuclide Results (picoCuries per gram [pCi/g])										
Sample	Radium-226	Radium-228 <sup>1</sup>	Combined Radium <sup>2</sup>	Thorium-230	Thorium-232	Combined Thorium <sup>3</sup>	Uranium-234	Uranium-235	Uranium-236	Uranium-238	Total Uranium <sup>4</sup>
1D7-84-85	3430	18.4 U	3430	57400	261	57661	203 D	9.33 D	0.0644 U	197 D	409
A1-AC-1	4574	24.7 U	4574	58800	119	58919	196 D	9.02 D	0.0612 U	197 D	402
A1-AC-3	1801	15.8 U	1801	38200	60.8	38261	92 D	4.49 D	0.0609 U	97.9 D	194
A2-AC-21	196	4.62 U	196	6680	9.85	6690	800 D	38.1 D	0.0897	816 D	1654
A2-S1	2514	20 U	2514	51800	118	51918	155 D	7.3 D	0.0649 U	155 D	317
A2-S1 (Duplicate)	NA	12.9 U	NA	50200	190	50390	164 D	7.63 D	0.0631 U	166 D	338
WL-234CT	1210	6.83 U	1210	26200	48.7	26249	68.7 D	3.34 D	0.0643 U	73.1 D	145

Notes

D Result is reported from a dilution

NA U Not analyzed

Indicates a non-detected result (result is less than the sample detection limit)

<sup>1</sup> As determined by activity of actinium-228.

<sup>2</sup> "Combined radium," a site-specific parameter used to identify radiologically-impacted material, is the sum of the radium-226 and radium-228 concentrations.

<sup>3</sup> "Combined thorium," a site-specific parameter used to identify radiologically-impacted material, is the sum of the thorium-230 and thorium-232 concentrations.

<sup>4</sup> "Total uranium" is the sum of all uranium isotope concentrations.

#### TABLE B-2

Sample		Isotopic	Uranium Results	(mg/kg)	Isotopic Composition (percent [%] by weight) <sup>1</sup>				
Sampie	Uranium-234	Uranium-235	Uranium-236	Uranium-238	Uranium, Total	Uranium-234	Uranium-235	Uranium-236	Uranium-238
1D7-84-85	0.0322	4.24	< 0.000990	580	584	0.0055%	0.73%	ND	99.3%
A1-AC-1	0.0311	4.10	< 0.000941	580	584	0.0053%	0.70%	ND	99.3%
A1-AC-3	0.0146	2.04	< 0.000937	288	290	0.0050%	0.70%	ND	99.3%
A2-AC-21	0.127	17.3	0.00138	2400	2410	0.0053%	0.72%	0.000057%	99.6%
A2-S1	0.0246	3.32	< 0.000999	456	460	0.0053%	0.72%	ND	99.1%
A2-S1 (Duplicate)	0.0261	3.47	< 0.000971	487	490	0.0053%	0.71%	ND	99.4%
WL-234CT	0.0109	1.52	< 0.000989	215	216	0.0050%	0.70%	ND	99.5%
Natural uranium <sup>2</sup>				0.0050%	0.72%	trace <sup>3</sup>	99.3%		

Notes

mg/kg Milligrams per kilogram

ND Not detected

<sup>1</sup> Isotopic Composition [percent by weight] = Concentration of uranium isotope in sample [mg/kg] / Concentration of total uranium in sample [mg/kg] x 100 percent

<sup>2</sup>Regarding the natural abundance of U-234, U-235, and U-238, see http://www.nrc.gov/reading-rm/basic-ref/glossary/natural-uranium.html and http://hps.org/documents/uranium\_fact\_sheet.pdf.

<sup>3</sup> Regarding the trace natural and anthropogenic abundance of U-236, see P. Steier et al., Nuclear Instruments and Methods in Physics Research Section B, May 2008.

#### TABLE B-3

## **RESULTS OF RADON EMANATION COEFFICIENT STUDY**

Sample and Bulk Ra-226 Activity	Test Condition	Percent Solids, as tested (wt %)	Percent Moisture, as tested (wt %)	Ra-226 (pCi/g)	Equilibrium Rn-222 (pCi/g)	Radon Emanation Coefficient
	1	81.06	18.94	3,900	547	0.14
	1 Dup	81.06	18.94	3,760	520	0.138
	2	99.72	0.28	3,787	195	0.0514
	3	100.00	0.00	4,333	178	0.0411
	3 Dup	100.00	0.00	4,563	191	0.042
<u>1D7-84-85</u>	4	100.00	0.00	4,235	155	0.0366
	5	80.94	19.06	3,994	359 J	0.0898 J
3,430 pCi/g	5 Dup	79.68	20.32	3,547	606 J	0.171 J
	6	90.21	9.79	4,270	571	0.134
	7	70.99	29.01	4,248	570	0.134
	8	98.18	1.82	4,229	301	0.0712
	8 Dup	98.80	1.20	3,880	242	0.0623
	9	91.81	8.19	3,900	211	0.0541
	1	85.05	14.95	5,253	674	0.128
	2	99.98	0.02	5,610	215	0.0383
	3	100.00	0.00	5,299	239	0.0452
<u>A1-AC-1</u>	4	100.00	0.00	5,652	186	0.033
	5	85.14	14.86	5,157	675	0.131
4,574 pCi/g	6	89.73	10.27	5,381	645	0.12
	7	73.99	26.01	4,928	583	0.118
	8	97.80	2.20	5,183	371	0.0716
	9	93.30	6.70	5,039	498	0.0989
	1	86.33	13.67	2,560	358	0.14
	2	99.80	0.20	2,524	133	0.0525
	3	100.00	0.00	3,013	138	0.0459
<u>A1-AC-3</u>	4	100.00	0.00	2,312	101	0.0435
	5	86.14	13.86	2,410	395	0.164
1,801 pCi/g	6	89.56	10.44	2,474	344	0.139
	7	70.42	29.58	2,425	391	0.161
	8	99.34	0.66	2,460	161	0.0654
	9	93.38	6.62	2,156	308	0.143
	1	81.35	18.65	249	118	0.473
	2	100.00	0.00	254	57.9	0.228
	3	100.00	0.00	296	36.7	0.124
<u>A2-AC-21</u>	4	100.00	0.00	299	38.9	0.13
106 . 61/	5	81.36	18.64	242	91.1	0.376
196 pCi/g	6	89.88	10.12	229	76.9	0.336
	7	67.22	32.78	237	84.6	0.358
	8	98.85	1.15	441	65.7	0.149
	9	89.34	10.66	237	92.7	0.391

#### TABLE B-3 (Continued)

## **RESULTS OF RADON EMANATION COEFFICIENT STUDY**

Sample and Bulk Ra-226 Activity	Test Condition	Percent Solids, as tested (wt %)	Percent Moisture, as tested (wt %)	Ra-226 (pCi/g)	Equilibrium Rn-222 (pCi/g)	Radon Emanation Coefficient
	1	78.76	21.24	2,801	186	0.0664
	2	99.86	0.14	3,081	160	0.0519
	2 Dup	99.91	0.09	3,100	160	0.0517
	3	100.00	0.00	3,201	98.1	0.0306
	4	100.00	0.00	2,998	132	0.044
40.01	4 Dup	100.00	0.00	3,143	157	0.0499
<u>A2-S1</u>	5	76.21	23.79	2,552	360	0.141
2,514 pCi/g	6	89.53	10.47	3,276	454 J	0.139 J
	6 Dup	90.06	9.94	2,923	181 J	0.0619 J
	7	68.46	31.54	2,692	393	0.146
	7 Dup	65.67	34.33	2,745	388	0.141
	8	99.32	0.68	2,844	179	0.0628
	9	91.47	8.53	2,764	334	0.121
	9 Dup	91.22	8.78	2,640	367	0.139
	1	73.37	26.63	1,600	217	0.136
	2	99.68	0.32	2,219	121	0.0547
	3	100.00	0.00	2,127	92.5	0.0435
<u>WL-234CT</u>	4	100.00	0.00	3,037	87.3	0.0287
	5	74.00	26.00	3,566	324	0.0907
1,210 pCi/g	6	89.92	10.08	3,541	317	0.0894
	7	59.13	40.87	2,845	357	0.126
	8	98.01	1.99	2,125	49.9	0.0235
	9	87.27	12.73	2,803	300	0.107

## Treatment Descriptions

Test Condition	Thermal Treatment	Moisture Treatment	
1	None (as received)	None. Samples maintained at their "as received" moisture content	
2	Heated at 105 °C for 16 hours	As found after thermal treatment (near 0 wt % moisture content)	
3	Heated at 250 °C for 16 hours	As found after thermal treatment (near 0 wt % moisture content)	
4		"As Found" after thermal treatment	
5	Heated at 250 °C for 48 hours	Moisture content adjusted to approximate the "as received" moisture content	
6		Water added to bring moisture content of sample to approximately 10 wt %	
7	None (as received) Water added to bring sample to a saturated moisture content		
8	Minimal heating at 60 °C to	ng at 60 °C to Water added and then dried until moisture content was approximately 2 wt %	
9	achieve desired moisture content	Water added and then dried until moisture content was approximately 10 wt $\%$	

## Notes

°C	Degrees Celsius	Ra-226	Radium-226
Dup	Laboratory duplicate	Rn-222	Radon-222
J	Data qualifier indicating the result is estimated	wt %	Percent by weight
pCi/g	picoCuries per gram		

**APPENDIX C** 

PLOTS OF RADON EMANATION COEFFICIENT

# Exhibit C-1





Test Cond	lition	Therma

- 1 None (as received)
- 2 Heated at 105 °C for 16 hours
- 3 Heated at 250 °C for 16 hours
- 4 Heated at 250 °C for 48 hours
- 5 Heated at 250 °C for 48 hours
- 6 Heated at 250 °C for 48 hours
- 7 None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

#### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content

Water added and then dried until moisture content was approximately 2 wt % Water added and then dried until moisture content was approximately 10 wt %

# Exhibit C-2





Test Condition	T

- Thermal Treatment None (as received) 1 2 Heated at 105 °C for 16 hours
- 3 Heated at 250 °C for 16 hours
- 4
- Heated at 250 °C for 48 hours
- 5 Heated at 250 °C for 48 hours 6 Heated at 250 °C for 48 hours
- 7
- None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

#### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content

Water added and then dried until moisture content was approximately 2 wt % Water added and then dried until moisture content was approximately 10 wt %




Test	Condition	Ther

Condition	<u>Thermal Treatment</u>
1	Nama (an manaimad)

- None (as received) 1 2 Heated at 105 °C for 16 hours
- 3 Heated at 250 °C for 16 hours
- Heated at 250 °C for 48 hours 4
- 5 Heated at 250 °C for 48 hours
- Heated at 250 °C for 48 hours 6
- 7 None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

#### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content





Test Condition	Thermal Trea

Condition	Thermal Treatment
1	None (as received)

- 2 Heated at 105 °C for 16 hours
- 3 Heated at 250 °C for 16 hours
- Heated at 250 °C for 48 hours 4
- 5 Heated at 250 °C for 48 hours
- Heated at 250 °C for 48 hours 6
- 7 None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

#### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content





Test Condition	Therma

- al Treatment None (as received) 1
- 2 Heated at 105 °C for 16 hours
- 3 Heated at 250 °C for 16 hours
- 4 Heated at 250 °C for 48 hours
- 5 Heated at 250 °C for 48 hours
- 6 Heated at 250 °C for 48 hours
- 7 None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

#### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content





Test Condition	Thermal

- l Treatment
- 1 None (as received) 2 Heated at 105 °C for 16 hours
- 3
- Heated at 250 °C for 16 hours
- 4 Heated at 250 °C for 48 hours
- 5 Heated at 250 °C for 48 hours 6 Heated at 250 °C for 48 hours
- 7
- None (as received)
- 8 Minimal heating at 60 °C to achieve desired moisture content
- 9 Minimal heating at 60 °C to achieve desired moisture content

### Moisture Treatment

None. Samples maintained at their "as received" moisture content As found after thermal treatment (near 0 wt % moisture content) As found after thermal treatment (near 0 wt % moisture content) "As Found" after thermal treatment

Moisture content adjusted to approximate the "as received" moisture content Water added to bring moisture content of sample to approximately 10 wt % Water added to bring sample to a saturated moisture content

APPENDIX D

**DATA VALIDATION REPORT** 

# Tetra Tech, Inc. DATA VALIDATION REPORT LEVEL IV

Site:	West Lake Landfill Site, Bridgeton, Missouri
Laboratory:	Southwest Research Institute (SwRI). (San Antonio, Texas)
Data Reviewer:	Harry Ellis, Tetra Tech, Inc. (Tetra Tech)
Review Date	May 4, 2016
Sample Delivery Group (SDG):	588049
Sample Numbers:	1D7-84-85, A2-S1, A1-AC-1, A1-AC-3, A2-AC-21, and WL-234CT
Matrix / Number of Samples:	Six Landfill Samples

The data were qualified according to the U.S. Environmental Protection Agency (EPA) Region 7 documents entitled "Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review" (9355.0-131), August 2014. In addition, the Tetra Tech document "Review of Data Packages from Subcontracted Laboratories" (February 2002), the project-specific "Quality Assurance Project Plan (QAPP) for Radon Emanation Coefficient Study" dated 29 October 2015, and the EPA and others document "Multi-Agency Radiological Laboratory Analytical Protocols Manual" (July 2004) were used along with other criteria specified in the applicable methods.

The review was intended to identify problems and quality control (QC) deficiencies that were readily apparent from the summary data package. The following sections discuss any problems or deficiencies that were found, and data qualifications applied because of non-compliant QC. The data review was limited to the available field and laboratory QC information submitted with the project-specific data package.

I, Harry Ellis, certify that all data validation criteria outlined in the above-referenced documents were assessed, and any qualifications made to the data accorded with those documents.

ang N. Elis

4 May 2016

Certified by Harry Ellis, Chemist

Date

# **DATA VALIDATION QUALIFIERS**

- U The analyte was not detected above the reported sample quantitation limit.
- **J** The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- UJ The analyte was not detected above the reported sample quantitation limit, which is estimated.
- **R** The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. Presence or absence of the analyte cannot be verified.

# DATA ASSESSMENT

Sample delivery group (SDG) 588049 included six (6) environmental landfill samples and no QC samples. Samples were analyzed by (1) thermogravimetric analysis (TGA) to determine the effects of heating, (2) inductively-coupled plasma (ICP) mass spectroscopy (MS) for uranium isotopes, (3) ICP atomic emission spectroscopy for non-radioactive metals, (4) alpha spectroscopy for thorium isotopes, (5) gamma spectroscopy for various thorium and uranium decay products, and (6) radon emanation coefficients. SwRI used their own methods that, where practical, followed the equivalent EPA SW-846 Methods. The following summarizes the data validation that was performed. Note that all holding time and chain of custody requirements were met.

# THERMOGRAVIMETRIC ANALYSES

# I. Method Description and Specific Objectives

Thermogravimetric analysis involves weighing a sample and heating it at a specified temperature for a specified period of time. During (and for while after) the heating period the sample weight is monitored and suitable thermal detectors are used to determine the presence of endothermic or exothermic reactions. The temperature versus time curve is useful for indicating reactions occurring within the sample. Therefore this technique is frequently used with known or presumed reactive materials, including flammable and explosive materials. For these tests, thermogravimetric analyses were used to prepare subsamples (called "conditions") for radon emanation tests as well as to determine moisture levels. Therefore few QC measures are taken.

II. Overall Assessment of Data

The analyses were performed as required in the QAPP. The only effect obvious from the results was evaporation of sample water, leaving a final residue with a mass of 60 to 86 percent of the original, wet sample.

# **ICP-MASS SPECTRAL ANALYSES**

I. Method Description and Specific Objectives

These analyses were a variant of the standard EPA SW-846 6020 analyses, modified to measure uranium isotopes (234, 235, 236, and 238, with uranium-233 added as an internal standard) only. It was used to characterize the uranium content of the samples.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS and sample duplicate analyses were performed on sample A2-S1. Recoveries could not be determined for isotopes (except uranium-234) because the unspiked sample contained much more of the isotope than the spike. No qualifications were applied for these data gaps. The sample duplicate analysis was within QC limit, so no qualifications were applied.

# III. Blanks

The laboratory (method) blank yielded no detectable concentrations of the analytes. No qualifications were applied.

IV. Laboratory Control Sample (LCS)

All percent recoveries from the LCS analyses were within established control limits. No qualifications were applied.

# V. Surrogates

Surrogates are not used in this analysis. Internal standard recoveries were well within their QC limits. No qualifications were applied.

VI. Other Quality Control Measures.

All calibration (initial, continuing, and low-concentration continuing) results were within their various QC limits. There were no irregularities with instrument performance checks, interference check samples, and the serial dilution analysis performed on sample A2-S1. No qualifications were applied.

VII. Comments

All analytes except uranium-236 in all samples were analyzed at dilutions, from 2- to 1,000-fold, to bring the results within calibration range. All samples were "natural uranium", with 0.7 percent uranium-235 and negligible uranium-236.

VIII. Overall Assessment of Data

Overall data quality is acceptable, with no qualifications applied. All data are usable as reported for their intended purposes.

# ICP-ATOMIC EMISSION SPECTROSCOPY ANALYSES

# I. Method Description and Specific Objectives

These analyses were essentially identical to the standard EPA SW-846 Method 6010 analyses, intended to characterize the samples for their contents of numerous metals, from aluminum to zirconium alphabetically and from lithium to bismuth in mass.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MSMSD and sample duplicate analyses were performed on sample A2-S1. Spike recoveries could not be determined for aluminum, calcium, cobalt, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, vanadium, and zinc because the field sample concentrations were much higher than the spike concentrations. No qualifications were applied for these data gaps. However, lanthanum recoveries were 59 and 46 percent, well below their QC limits of 75 to 125 percent. The lanthanum results for all samples were qualified as estimated, possibly biased low, and flagged "J". In addition, the only result outside the QC limit of an RPD of 20 percent for the sample duplicate analysis was for tin, with an RPD of 30.5 percent, indicating a heterogeneous distribution of the metal. Due to this uncertainty, the result for tin in sample A2-S1 was qualified as estimated and flagged "J".

# III. Blanks

The laboratory (method) blank yielded no reportable concentrations of the analytes. No qualifications were applied.

IV. Laboratory Control Sample (LCS)

The LCS included two standard reference materials (basalt rock and obsidian rock) from the National Institute of Standards and Technology (NIST), plus the customary laboratory-generated ones. All percent recoveries from all LCS analyses were within established control limits. No qualifications were applied.

# V. Surrogates

Surrogates are not used in this analysis. Internal standard recoveries wee within established control limits. No qualifications were applied.

# VI. Other Quality Control Measures

Almost all (initial, continuing, and low-concentration continuing) calibration results were within their various QC limits. The only exceptions were the low-concentration continuing calibration results for boron, which exceeded QC limits with concentrations of 201 to 205 percent. Boron was not detected in any field samples, so no qualifications were applied. In addition, there were no irregularities with interference check samples and the serial dilution performed on sample A2-S1. Again, no qualifications were applied.

# VII. Comments

Many analytes in all field samples were reported from re-analyses at 10- or 100-fold dilutions, to bring the results within linear calibration range and minimize matrix interference. No qualifications were applied.

VIII. Overall Assessment of Data

Overall data quality is acceptable, with no significant qualifications applied. All data are usable as qualified for their intended purposes.

# ALPHA SPECTROSCOPY ANALYSES

#### I. Method Description and Specific Objectives

These analyses, using standard alpha spectroscopy methods are intended to help characterize the thorium isotopes (228, 230, and 232) in the field samples.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD analyses were not performed. The sample duplicate analysis was performed on sample A2-S1 and yielded satisfactory results. No qualifications were applied.

#### III. Blanks

The laboratory (method) blank yielded low activities for thorium-228 and -230. Field sample results for thorium-230 (a daughter of uranium-238) were considerably higher than the blank concentration, so no qualifications were applied. However the field sample results for thorium-228 (a daughter of thorium-232) were only slightly greater than the blank. Therefore the field sample results for thorium-228 were qualified as estimated, possibly biased high, and flagged "J".

IV. Laboratory Control Sample (LCS)

All percent recoveries from the LCS analyses were within established control limits. No qualifications were applied.

#### V. Surrogates

Surrogates are not used in this method. A "tracer" if thorium-229 was included in sample preparation and provides the same information as a surrogate. All recoveries were within QC limits so no qualifications were applied.

VI. Comments

Calibration results were within their QC limits.

VII. **Overall Assessment of Data** 

Overall data quality is acceptable, with no significant qualifications applied. All data are usable as qualified for their intended purposes.

# GAMMA SPECTROSCOPY ANALYSES

#### I. Method Description and Specific Objectives

Gamma spectroscopy was used to characterize a variety of daughters of the naturally occurring uranium and thorium already discussed. The method is the standard one of putting a sample in an air-tight can (to 103I9025140007 6 SDG 588049

retain radon until it decays) and placing that in a suitable counting instrument. In these reports, total propagated uncertainty (TPU) is given as the "one-sigma" level (range of 68 percent about the reported activity) instead of the "two-sigma" results (range of 95 percent) more generally used.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

No MS/MSD analyses were included. Laboratory duplicate analysis yielded results well within QC limits. No qualifications were applied.

III. Blanks

The laboratory blank yielded no detectable analyses. No qualifications were applied.

IV. Laboratory Control Sample (LCS)

All percent recoveries from the LCS analysis were within established control limits. No qualifications were applied.

V. Surrogates

Surrogates are not used in this radioanalytical method.

VI. Comments

Calibration results were within their QC limits.

VII. Overall Assessment of Data

Overall data quality is acceptable, with no qualifications applied. All data are usable as reported for their intended purposes.

# **RADON EMANATION ANALYSES**

I. Method Description and Specific Objectives

SwRI used 1-liter gas-tight "emanation chambers" for testing nine portions of each field sample after appropriate heat treatment and, if called for, re-hydration. After a suitable ingrowth period (5 days or more), each emanation chamber was connected to a 98-milliliter "Lucas Cell", which has a lining that flashes when alpha radiation hits it. Gamma spectroscopy was also used to determine some isotopes. The Lucas Cell is a standard technique for determining radon emanations from various materials, including drinking water and mine tailings. Determination of these radon emanation coefficients (the equilibrium radon activity divided by the radium-226 activity) under the various "conditions" is the primary objective of the study.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

No MS/MSD analyses are included. No qualifications were applied for the data gap. Most laboratory duplicate results were well within QC limits. The exceptions were Condition 5 (heated at 250 °C for 48 hours) for sample ID7-84-85 and Condition 6 (Condition 5 followed by re-hydration to 10 percent moisture) for sample A2-S1. Those two results are qualified as estimated, apparently due to heterogeneity in the field samples, and flagged "J". Similar irregularities may exist in non-tested samples.

III. Blanks

The laboratory (method) blank yielded no detectable analytes. No qualifications were applied.

IV. Laboratory Control Sample (LCS)

All percent recoveries from the LCS analyses were within established control limits. LCS were created from both a "radon generator" designed to interface directly with a Lucas Cell and with capsules of a suitable solid-phase standard placed in an emanation cell and treated as a field sample. No qualifications were applied.

# V. Surrogates

Surrogates are not used in these radioanalytical methods.

VI. Comments

None.

VII. Overall Assessment of Data

Overall data quality is acceptable, with no significant qualifications applied. All data are usable as qualified for their intended purposes.