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Analysis of Soluble Rhenium Concentrations in the Refractory from Bulk Vitrification Full-Scale Test 38B

S.K. Cooley
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June 2006



Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Summary

The Hanford Site's radioactive tank waste resulted from more than 40 years of nuclear materials production. The capacity of the waste treatment plant is not sufficient to process all of the tank waste and support the Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement) milestone to complete tank waste treatment by 2028. Therefore, a cooperative approach led to the selection of bulk vitrification as a possible way to accelerate tank waste treatment by providing some supplemental low-activity waste (LAW) treatment capacity.

Bulk vitrification combines LAW and glass-forming chemicals within a large metal disposal container (often referred to as the box) and melts the contents using electrical resistance heating. A castable refractory block (CRB) is used along with sand to insulate the metal container from the heat generated while melting the contents into a durable glass waste form. Engineering-scale (ES) and full-scale (FS) tests have been conducted as part of the development and qualification of the bulk vitrification process. Several ES tests conducted to evaluate the bulk vitrification process showed that a small fraction of soluble technetium (^{99}Tc) moves into the CRB and is projected to result in a groundwater peak different than that in glass from the Waste Treatment Plant. The total soluble ^{99}Tc fraction in the FS box is expected to be different than that determined from the ES tests. However, until FS tests are available, the best-estimate soluble ^{99}Tc fraction from the ES tests has been used as a conservative estimate for the full-size system.

The first FS test results available for evaluation are from cold simulant tests that have been spiked with rhenium (Re). Although definitive information will not be available until FS tests with ^{99}Tc have been conducted, an approach can be applied to estimate a scale-up factor that extrapolates the ^{99}Tc data collected at the ES to the FS bulk vitrification waste package. FS-38A was run to test the refractory ^{99}Tc design and did not have a Re spike. Samples from this test were taken and analyzed to help determine CRB extractable Re background concentrations. FS-38B used a Re-spiked, six-tank composite, dry waste simulant that was mixed with soil and glass formers to produce the waste feed. Although this dry feed is not physically the same as the Demonstration Bulk Vitrification System feed that will be prepared by drying a solution of actual waste constituents, the chemical make-up is the same. Extensive sampling of the CRB from test FS-38B was planned, but difficulties with the test prevented filling the box with the desired quantity of glass. Based on the nonprototypic nature of FS-38B, an abbreviated plan was devised that examined duplicate samples from refractory archive sections, a lower wall sample, and two base samples to gain early information related to Re and projected ^{99}Tc levels in the full-scale box.

Although the FS-38B tests did not generate a full box of glass, the abbreviated sampling plan was useful for two reasons. First, the analysis of the FS samples clarified the sampling and analysis techniques that would obtain the information needed from the full analysis planned in test FS-38C. Second, these limited analyses pointed out several differences between ES and FS test results and were useful in advancing the understanding of Re/Tc migration into the CRB at full scale. The key conclusions and recommendations from the FS-38B CRB analyses are listed below.

Conclusions

- The CRB material contains a measurable extractable Re background level. This level is highly variable, ranging from 1 µg/kg, which does not require background adjustments, to more than 40 µg/kg, which requires background adjustments to correctly interpret Re results.
- Estimates of the extractable Re fraction in the FS-38B CRB ranged from 0.75% with no background adjustment to 0.52% for a background adjustment of 40 µg/kg.
- The initial FS-38B results indicate that a smaller fraction of the Re present in the feed moves into the CRB at FS than at ES. The ES/FS scale-up factor was fairly independent of the background adjustment levels and ranged from 2.83 to 2.65.
- ⁹⁹Tc fractions in the CRB projected from FS-38B Re results ranged from 0.13 to 0.21% depending on the correction for extractable Re background level.
- The two segments used to analyze the extractable Re concentration in each refractory sample indicated that Re penetrates deeper into the FS CRB than into the ES CRB.
- Duplicate samples from same locations on the CRB showed reasonably good agreement in the concentrations of extractable Re.
- The two base samples showed significantly different results. This demonstrates the need to collect multiple samples from each vertical section of the CRB to assess the variability of Re concentrations in different parts of the box and to obtain a more reliable estimate of extractable Re present.
- Although the abbreviated CRB sampling routine used in FS-38B did not allow examination of horizontal concentration effects, the limited data did indicate a significant vertical effect with a substantial concentration of extractable Re in the CRB at the melt line.
- Assumptions about affected volumes and volume uncertainty did not have a significant impact on the amount or uncertainty of extractable Re estimates.

Recommendations

- The FS CRB samples taken for Re analysis must meet specifications to be useful. It is recommended that personnel familiar with the refractory analysis process be present during CRB sampling. The CRB should be retained and protected from the environment until all samples have been transferred and approved by the analyzing organization.
- The deeper penetration of Re into the FS CRB indicates that Re levels in the sand between the CRB and the bulk vitrification container should be measured in subsequent FS tests. The extractable Re background level of the sand should also be determined as part of the sand testing.
- Analyzing the CRB samples as inner and outer segments provided useful information about Re distribution in the FS CRB. Although combining the solutions produced for each sample would reduce the number of inductively coupled plasma-mass spectrometry samples by half, it is recommended that the solutions continue to be analyzed separately to monitor penetration levels.
- Future FS tests should continue to investigate spatial effects on Re deposition in the CRB. The potential for significant vertical and horizontal effects should be explored.

- The uncertainty associated with the extractable Re background level should be minimized by analyzing multiple blank samples of the FS-38C CRB material.
- The variable extractable Re background indicates that any future ES tests should use larger Re spike levels to reduce the effect of CRB extractable Re background levels on the final results.

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1.0 Introduction

1.1 Background

The Hanford Site's radioactive tank waste resulted from more than 40 years of nuclear materials production. Baseline plans for disposition of these wastes include the separation of low-activity (LAW) and high-level (HLW) fractions of the waste, followed by vitrification of both fractions to produce immobilized LAW and HLW glass forms. The immobilized HLW will be disposed in a deep geologic repository, while the immobilized LAW will be disposed in a shallow burial facility at Hanford. Separation and vitrification operations will be performed in the Waste Treatment Plant (WTP) that is currently under construction on the Hanford Site. However, the capacity of the WTP is not sufficient to process all of the tank waste and support the Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement) (TPA) milestone to complete tank waste treatment by 2028. Therefore, the U.S. Department of Energy (DOE) Office of River Protection, Washington State Department of Ecology, and the U.S. Environmental Protection Agency agreed to cooperate in developing approaches to accelerate tank waste treatment by providing some supplemental LAW treatment capacity (DOE 2002).

After assessing dozens of technologies and testing and evaluating three preferred LAW immobilization approaches in 2002 and 2003, bulk vitrification was selected for a pilot supplemental treatment test and demonstration facility (Raymond et al. 2004). The goal is to further evaluate bulk vitrification through testing of both simulated and real waste to support the TPA milestone (M-62-11) associated with a final decision on treating the tank waste that is beyond the capacity of the WTP.

Bulk vitrification combines LAW and glass-forming chemicals within a large disposal container and melts the contents using electrical resistance heating. Bulk vitrification employs a disposable melter (a steel container, often referred to simply as a box) that is disposed along with the vitrified waste form in a LAW burial ground after the waste form has cooled. Because the bulk vitrification melter is used only once, some of the processing constraints of the baseline joule-heated, continually fed ceramic melters can be avoided. The in-container bulk vitrification process mixes and dries LAW, soil, and glass-forming chemicals and then melts the mixture at approximately 1250° to 1500°C by electrical resistance (Kim et al. 2003). Graphite flakes are added to the mix to form a conductive path to initiate melting. Electrical current is supplied through two graphite electrodes imbedded in the batch. The design concept for early testing used top-down melting of a large, single batch of waste and soil that was surrounded by an insulating primary liner of quartz sand to protect the steel container from the glass melt. The current design uses bottom-up melting where, after melt initiation of a small batch, waste is loaded incrementally until the container is filled with waste glass. A rigid castable refractory block (CRB), rather than quartz sand, is used as the primary liner to protect the steel container from the glass melt. A layer of sand is used as a secondary insulating layer between the CRB and the steel container.

Engineering-scale (ES) and full-scale (FS) tests have been conducted as part of the development and qualification of the bulk vitrification process. The current FS bulk vitrification design uses steel containers that are 2.3 m tall, 2.3 m wide, and 7.32 m long (7.5×7.5×24 ft). ES tests use a container that is about 1/6th linear scale relative to the FS melts. The ES tests are conducted to gather information on several process variables that cannot be obtained with crucible-scale tests. Both ES and FS steel containers use refractory materials to insulate the container walls from the high-temperature glass melt.

The glass melt is in direct contact with the CRB, which is composed of several precast refractory panels surrounded by quartz sand. A thin insulating board (i.e., duraboard) is also used between the ends of the steel container and CRB to allow for thermal expansion. The ES containers are lined with a 10.2-cm (4-in.) CRB wall, a 5.1-cm (2-in.) layer of quartz sand, and a 5.1-cm (2-in) layer of duraboard insulation. The FS containers are lined with a 15.2-cm (6-in.) CRB, 10.2 cm (4 in) of quartz sand, and a 5.1-cm (2-in) layer of duraboard insulation at the box ends. The FS containers also have refractory support blocks that help ensure that the precast refractory panels of the CRB stay in place. Figure 1 is a schematic of the FS refractory materials.

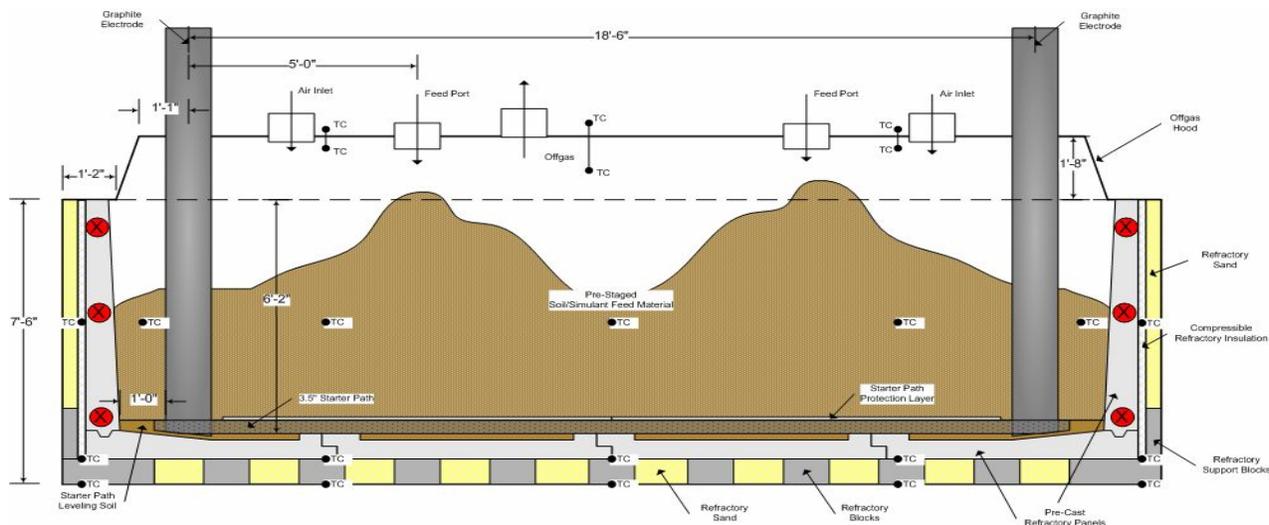


Figure 1. FS Refractory Materials

1.2 Previous Testing

Results of early ES and FS tests of the bulk vitrification process suggested that a small portion of the technetium (^{99}Tc) waste stream inventory would end up in a soluble form deposited in a vesicular glass layer at the top of the bulk vitrification melt and in the quartz sand insulation (McGrail et al. 2003, Thompson 2003). Based on an initial risk assessment, the small amount of soluble ^{99}Tc salt in the bulk vitrification waste package is projected to create a ^{99}Tc concentration peak at early times in the groundwater extracted from a well 100-m downgradient (Mann et al. 2003). This peak differed from the predicted baseline WTP glass performance, which showed an asymptotic rise to a constant release rate. Because regulatory agencies want supplemental treatment technologies to produce a product whose performance is essentially equal to that of WTP glass, the bulk vitrification process was modified to minimize deposition of soluble ^{99}Tc salts. Using a CRB in place of a portion of the refractory sand layer and a bottom-up melting technique reduced ^{99}Tc deposition in the porous refractory sand and eliminated the vesicular glass layer at the top of the melt.

Several ES tests were conducted to evaluate the modified bulk vitrification process and quantify the deposition of soluble ^{99}Tc or rhenium (Re), its nonradioactive surrogate. Results showed that the bottom-up melting eliminated the vesicular glass layer on the melt surface, and the CRB significantly reduced deposition in the refractory liner. Although significant reduction in the soluble ^{99}Tc fraction was achieved

through process modification, a small fraction of soluble ^{99}Tc still remained in the CRB and was projected to result in a groundwater peak different than WTP glass. The average best estimate of the ^{99}Tc inventory that would be leachable from the CRB in these ES tests is $0.37 \pm 0.12\%$. The best estimate for the Re levels in the CRB is $2.11 \pm 0.74\%$ (Pierce et al. 2006). To compare the results from multiple tests, the concentrations in the CRB are expressed as a fraction of the material batched into to feed.

The total soluble ^{99}Tc fraction in the FS box is expected to be different than that determined from the ES tests. Several factors in FS are different from ES, such as lower surface-area-to-volume ratio and lower power requirements per mass of feed melted, indicating that the soluble ^{99}Tc fraction in the large box may be significantly less. However, until FS tests are available, the best-estimate soluble ^{99}Tc fraction from the ES tests has been used as a conservative estimate for the full-size system.

The first FS test results available for evaluation are from cold simulant tests that have been spiked with Re. Although definitive information will not be available until FS tests with ^{99}Tc have been conducted, an approach similar to that used by McGrail et al. (2003) can be used to estimate a scale-up factor that extrapolates the ^{99}Tc data collected at the ES to the FS bulk vitrification waste package. This methodology uses a $^{99}\text{Tc}/\text{Re}$ mobility ratio determined from ES tests to adjust the Re levels measured in FS tests. Two ES tests that were spiked with both ^{99}Tc and Re showed that Re was more mobile, with an average $^{99}\text{Tc}/\text{Re}$ mobility ratio of 0.17 ± 0.05 (Pierce et al. 2006). This ratio is calculated by dividing the amount of the soluble ^{99}Tc in the CRB by the amount of soluble Re in the CRB when both are expressed as a fraction of the starting inventory. Multiplying the Re levels determined from FS tests by the $^{99}\text{Tc}/\text{Re}$ mobility ratio supplies an estimate of the anticipated ^{99}Tc concentrations that might be present at full scale.

FS-38A was run to test the refractory design and did not have a Re spike. Samples from this test were taken to determine Re blank levels. FS-38A1 was spiked with Re but did not use a waste simulant that contained nitrate and sulfate salts for six of the eight batches melted. Nitrate and sulfate salts are thought to be key components that melt at low temperatures to form a molten ionic salt (MIS) and carry Tc and Re into the CRB as the MIS penetrates the porous CRB (Bagaasen et al. 2006). FS-38B was the first FS test that could be used to estimate the amount of Re in the CRB. FS-38B used a six-tank composite dry waste simulant mixed with soil and glass formers to produce the waste feed. Although the dry feed is not physically the same as the Demonstration Bulk Vitrification System feed that will be prepared by drying a solution of waste constituents, the chemical make-up is the same. The amount of Re that is added to a FS test is controlled by monitoring the quantity of a concentrated contaminant of potential concern blend that is added to the melt. This blend is spiked with the required quantity of Re and analyzed to determine the actual concentration. The spiked and analyzed concentrations demonstrate good agreement, but the analyzed values are used in final calculations to account for any possible errors introduced by the purity of the spike material (e.g., difference in quantity of hydrated water).

Initially, extensive sampling of the CRB from FS-38B was planned (36 locations), but difficulties with the feed transfer and off-gas systems prevented completion of a full box. Based on the nonprototypic nature of FS-38B, an additional test (FS-38C) was planned. The extensive sampling of the CRB was moved to FS-38C, but an initial look at the FS-38B CRB results was thought to be useful. An abbreviated plan that looked at 10 samples was devised based on the nonprototypic box fill. This plan was again modified when sampling instructions were misinterpreted and the original refractory samples were not acceptable for analysis. Instead, samples were taken from refractory archive sections. The main

consequence of taking samples from the archived sample material was that the two samples taken from different wall heights were essentially duplicates taken from the same area; the original plan had called for two samples taken at the same height from two different areas of the CRB.

2.0 Description of the CRB

The FS CRB is made by casting vibrocast 60PC into several sections and assembling the sections in the bulk vitrification container. Vibrocast 60PC is a 60-percent alumina, mullite-based, low-cement castable designed to provide minimum porosity and low linear change with maximum density (2611 kg/m^3) and high fired strength. The material was chosen primarily for its ability to withstand severe high-temperature corrosive and erosive environments. Although the vibrocast material is dense by refractory standards, the material is still porous and has a high relative surface area (Figure 2).

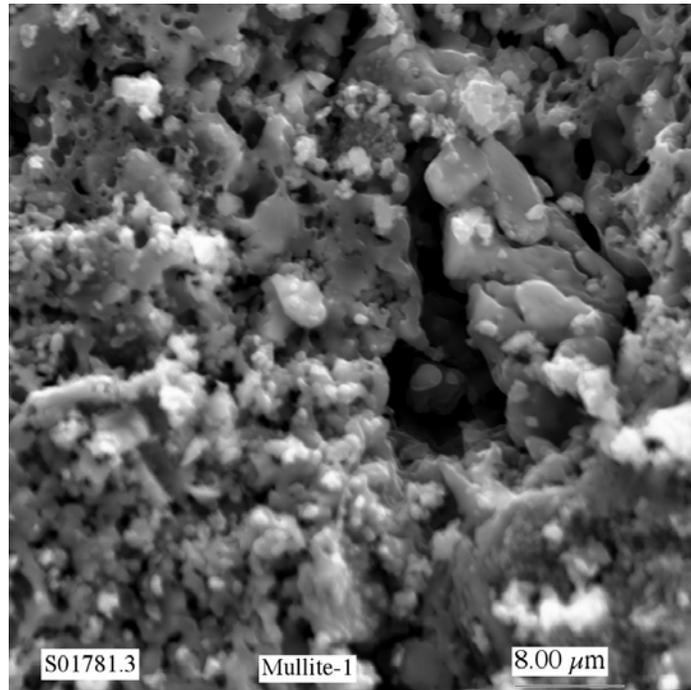


Figure 2. SEM Image of Castable Refractory

Based on past ES tests, the Re deposition in the CRB for FS-38B is expected to have a significant vertical effect; that is, the concentration of Re present in the CRB is expected to follow a vertical gradient. However, past ES tests do not suggest any significant horizontal effects, either along the length or along the width of the refractory. Therefore, calculations presented here involve vertical sections or layers, as depicted in Figure 3. For convenience, the vertical sections are often referred to simply as sections of the CRB. In general, there are three types of vertical sections: upper CRB wall sections, the bottom CRB wall section, and the CRB base. Additional details of the bulk vitrification container and the CRB are provided in AMEC ICV box refractory assembly drawings such as F-145579-35-D-0016 and F-145579-35-D-0004.

Sampling plans for bulk vitrification tests (both ES and FS) typically call for samples to be taken from specified locations around the CRB walls and base. In Figure 3, the potential wall sampling locations are indicated by an X within a red circle.

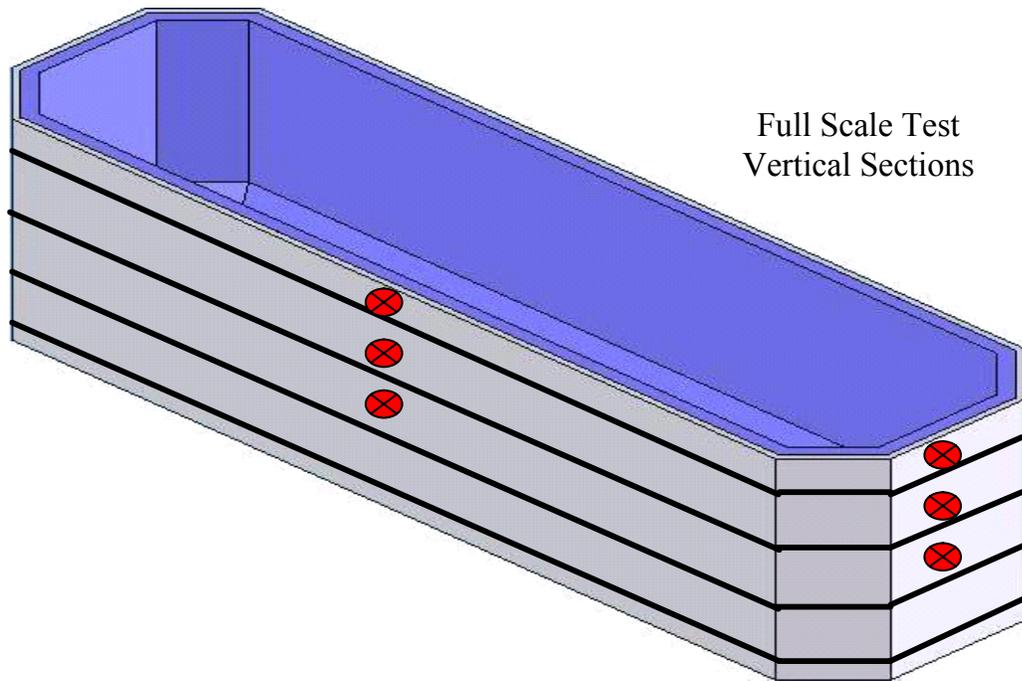


Figure 3. Vertical Sections of CRB for FS Test

2.1 Vertical Sections of FS-38B

FS-38B sampling was conducted using available/accessible portions of the CRB. Six CRB sections were defined as a result of the sampling process implemented for FS-38B. Sections 1 through 4 are upper wall sections, section 5 is the bottom section of the wall, and section 6 is the CRB base. Figure 4 depicts a cross-sectional view of the CRB wall and base with sections identified as defined for FS-38B. The simplifying assumptions for the CRB shape do not significantly affect the Re fraction or scale-up factor results.

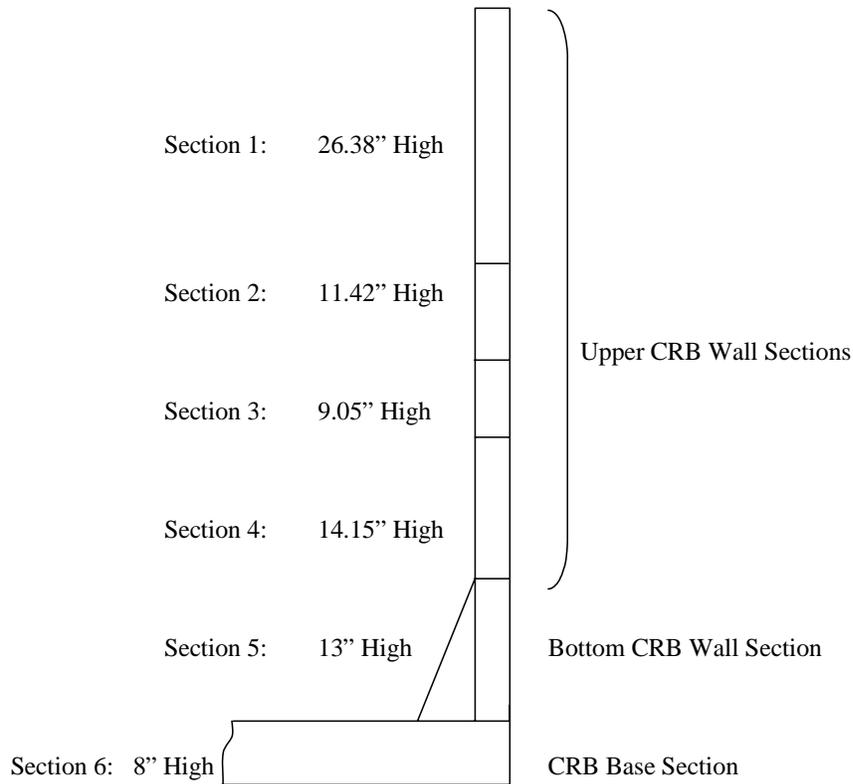


Figure 4. Illustration of CRB Wall and Base Sections for FS-38B

As shown in Figure 4, section 1 covers the top 67 cm (26.38 in.) of the wall, and section 2 covers the next 29 cm (11.42 in.) below that. Section 3 covers the next 23 cm (9.05 in.), and section 4 covers the remainder of the upper wall, approximately 155 cm (14.15 in.). Although the CRB wall is actually slightly tapered, calculations used in this report are based on the assumption that the walls are vertical. Thus, the upper wall sections are assumed to have rectangular-shaped cross sections. Section 5, which is the bottom section of the wall, has a chamfered shape, so the cross section can be described as a rectangular-shaped region together with a triangular-shaped region. Although the CRB base, section 6, actually has several sump regions, the calculations used in this report are based on the assumption that the base section is flat and has a uniform thickness.

Rhenium deposition (extractable Re mass) is estimated for each vertical section of the CRB and is a function of the volume, Re concentration, and density of each section. For estimating Re deposition in the CRB, the height of the upper wall sections can differ depending on actual sampling location. The number of upper wall sections defined can differ from one FS test to another as well. Regardless of their height, each upper wall section has the same general shape. Thus, in estimating Re deposition, the same general equation can be used to approximate the volume of each upper wall section. However, volume approximation calculations used to estimate Re deposition require the bottom wall section and base to be treated separately because of their different shapes. Accordingly, if sampling plans for future FS tests do not call for sampling from the bottom CRB wall section, Re concentrations from samples at the next higher wall section can be used to represent the concentration in the bottom wall section. It is expected that all future sampling plans will call for sampling from the CRB base.

3.0 CRB Samples for FS-38B

The techniques and procedures used to sample, analyze, and calculate the soluble fraction of Re in each CRB sample are described in Pierce et al. (2004). A brief summary of the techniques is provided in this section. Several samples were taken from large subsections of the FS CRB wall and base using an electric jackhammer and broken into fragments of the appropriate size for the equipment used in the extraction. Each sample had an approximate cross section of 2×2 inches and penetrated the full 6- to 8-inch thickness of the refractory wall, as illustrated in Figure 5.

Figure 5 depicts the approximate vertical locations for the wall sections of the CRB as well as one of the sample locations on the base. The shaded regions are divided into two subregions depicting the two fragments of each sample. In each pair, the fragment adjacent to the glass is referred to as the inner fragment, and that along the outer CRB surface (not in contact with the glass) is referred to as the outer fragment. The lowest wall sample was not divided into two segments.

Eleven CRB samples from FS-38B were analyzed. Eight consisted of duplicates taken from the vertical top, above the melt-line, at the melt-line, and below the melt line (see Figures 6 and 8). Two samples were taken from the base portion of the CRB (see Figure 7). Each sample was fractured into two roughly 3- to 4-inch-long fragments, as illustrated in Figure 5. The inner fragment represented the Re deposition in the CRB from the glass/refractory interface to the center of the CRB, while the outer segment represented the deposition from the center of the CRB to the refractory/sand interface.

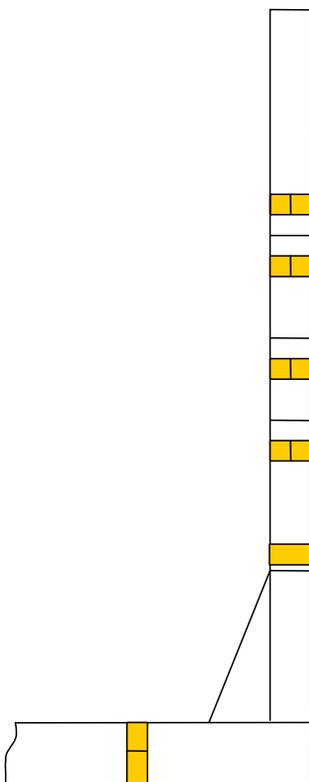


Figure 5. Side View of FS-38B CRB. Sample locations are depicted in gold; lines depict wall sections.

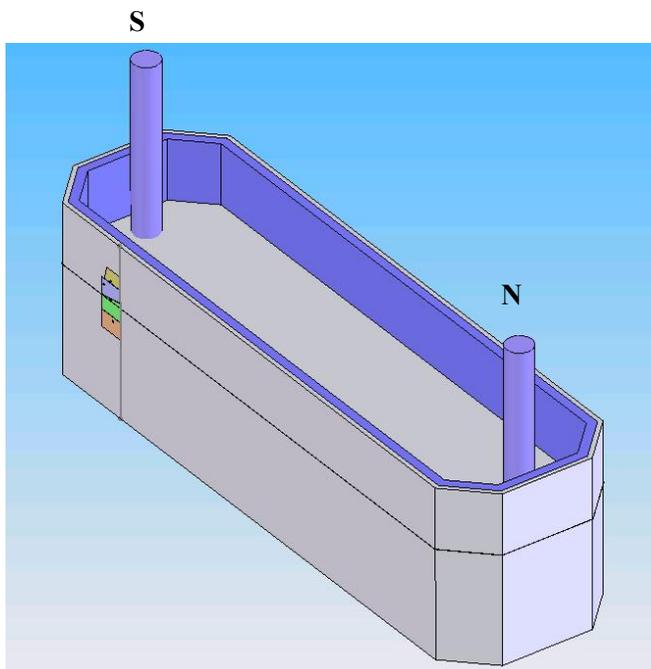


Figure 6. Isometric View of FS-38B CRB with Sample Locations Depicted. The horizontal line on the outer wall of the CRB represents the final melt height.

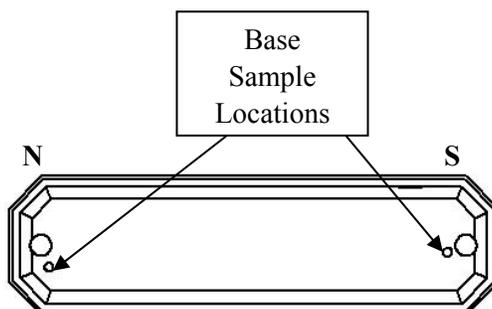


Figure 7. Base Sample Locations

One sample was taken from the lower wall section (see Figure 9). It was the best available sample from that section of the CRB and included portions that were in contact with the glass. However, the sample crumbled when it was removed from the CRB, and it was impossible to determine which fragments were associated with the inner and outer portions of the CRB, so only a total value is reported for this sample.

3.1 Sample Locations for FS-38B

Figures 6 through 9 illustrate the sample locations for FS-38B. The irregularly shaped regions shown in different colors represent large portions of the CRB wall that were taken as archive samples. The red dots (see Figure 8) represent the approximate location of the duplicate wall samples that were collected

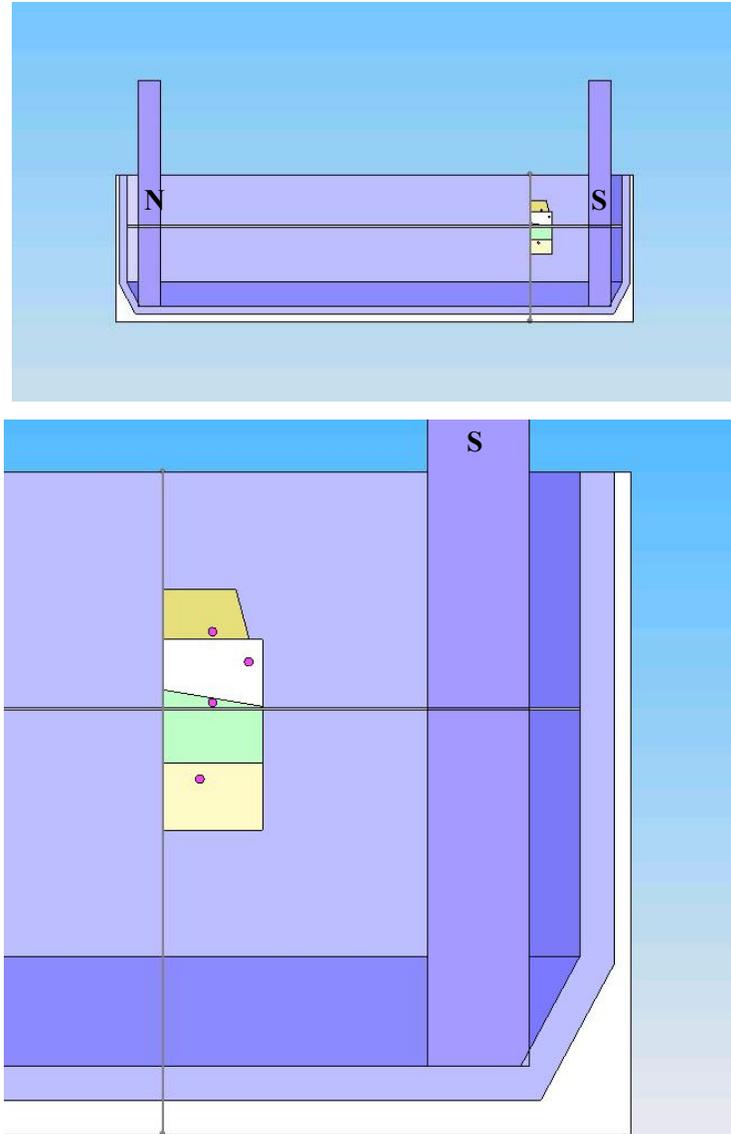


Figure 8. Full and Close-up Side View of FS-38B CRB Depicting Sample Locations. Sample locations are shown from the inner wall surface to illustrate final melt line level.

for analysis. In addition, two samples were taken from the CRB base, one near each of the two electrodes, as shown in Figure 7. The location of the lower wall sample from segment 5 is shown in Figure 9. The electrodes are labeled N and S to represent the north-south orientation of the bulk vitrification container for FS-38B.

Figure 10 shows the large backhoe operated jack hammer that was used to obtain the large CRB archives that were used to obtain the FS-38B CRB samples. Figures 11 and 12 show the large base and wall CRB portions prior to obtaining smaller samples with a hand held electric jack hammer. The arrow in Figure 10 identifies a vertical seam used to seal two wall panels of the CRB. This particular seam is identified because the upper wall samples from FS-38B were taken from portions of the CRB that bordered this seam. The seam is depicted as a vertical line in Figures 6, 8, and 9.

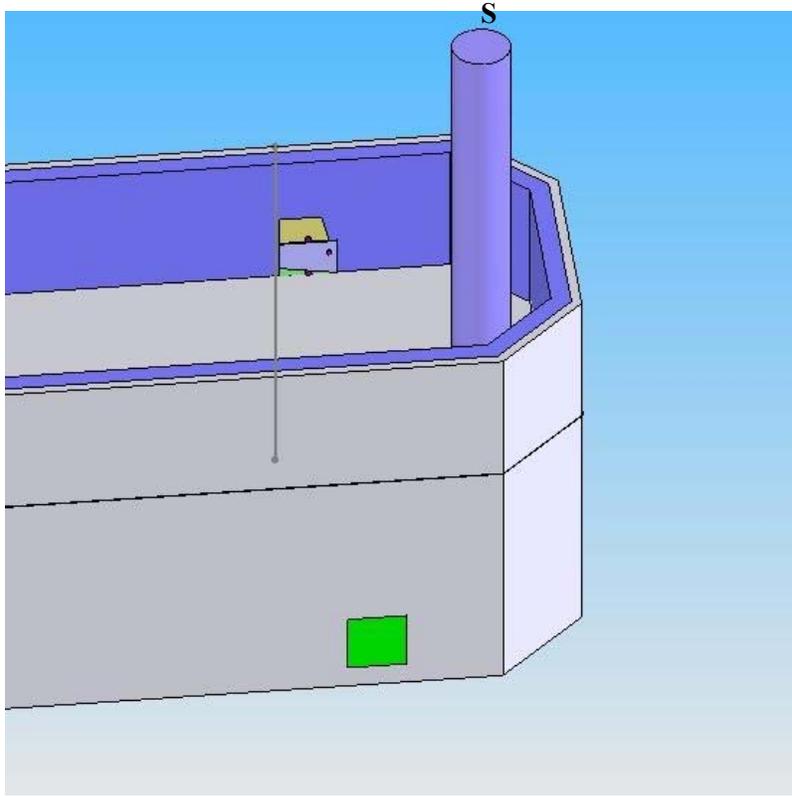


Figure 9. Approximate Location of the Section 5 Sample (green box)

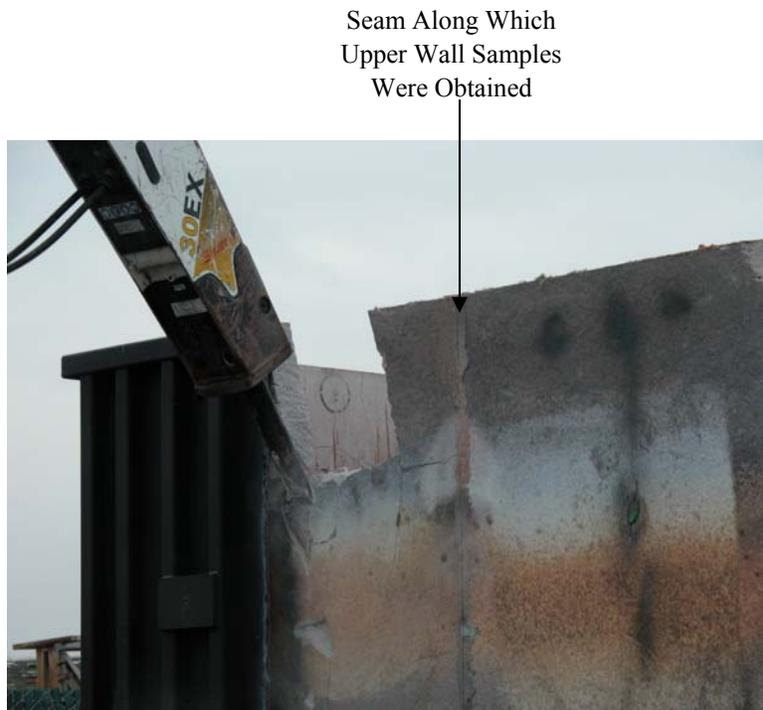


Figure 10. The Backhoe-Operated Jackhammer Obtains Large Archive Samples from the Southwest Side of the CRB Wall

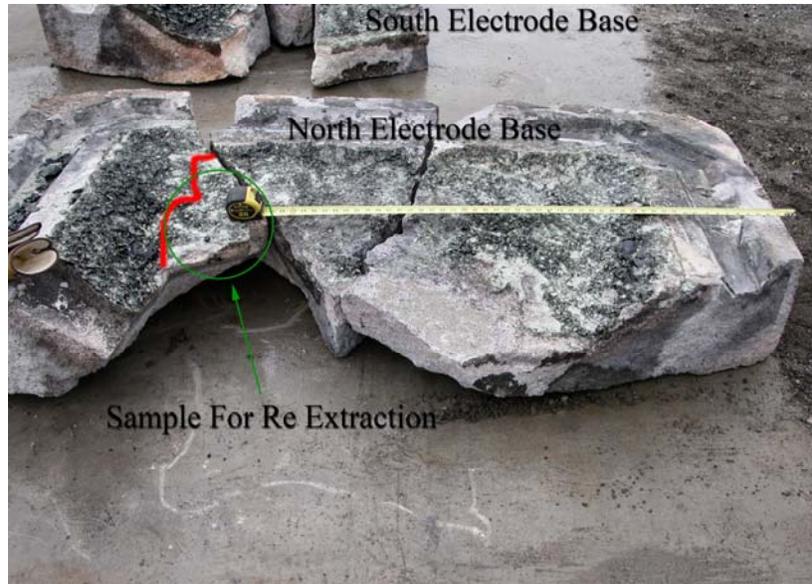


Figure 11. Base Sections Taken from North and South Electrodes Used for CRB Samples

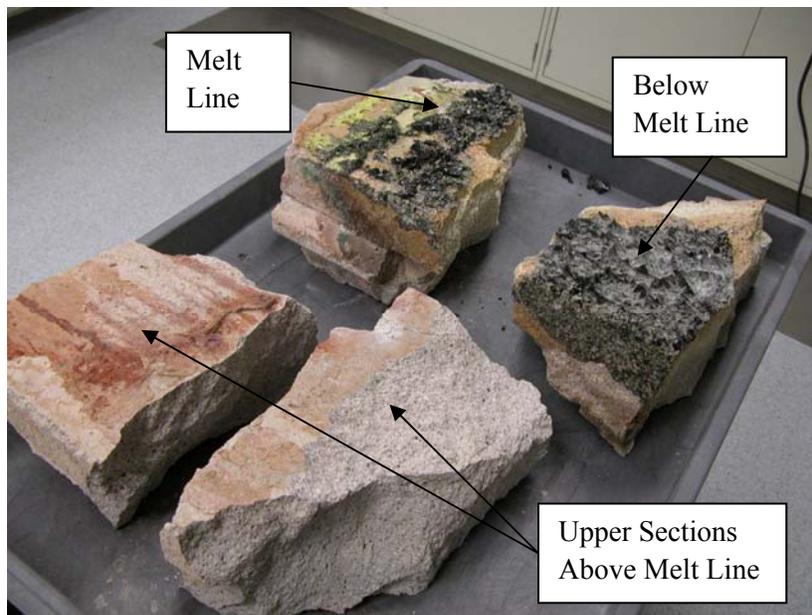


Figure 12. Wall Sections Used for CRB Samples

3.2 Limitations Related to Samples

Ideally, estimates of Re concentration and corresponding variability would be based on a sampling plan with adequate numbers of samples per CRB section and sampling locations that cover the different vertical sections. Such sampling plans generally lead to estimates of Re concentration and variability that accurately reflect within-sample variability and between-sample variability over a given CRB section.

This type of sampling is planned for FS-38C, but the number and type of samples available from FS-38B did not support such a sampling plan.

For FS-38B, samples were taken from a relatively small region of the CRB and therefore may not be representative of the CRB as a whole. Because of their proximity, the samples are essentially duplicates and thus provide a better estimate of within-sample variability than between-sample variability. The between-sample variability, or location-to-location variability, is likely to be greater than the within-sample variability. As a result, the samples from FS-38B may not adequately capture the actual variability in Re content that exists within the CRB sections and therefore across the entire CRB. Thus, it is likely that the FS-38B samples underestimate the uncertainty associated with Re concentration for the CRB sections.

Estimations of variability in the mean Re concentration for each section of the CRB depend on the number of samples from each section (see Equation A.8 in the appendix). Larger numbers of samples effectively reduce the estimates of variability in the mean Re concentration for each section. The limited number of samples taken from each section of FS-38B results in minimal effective reduction in the variance estimates. For section 5, where only one sample was available, it was impossible to calculate a standard deviation using the method of Equation (A.8), so the section standard deviation was estimated as the standard deviation calculated from the fragments of the sample. For this case, it is clear that the uncertainty estimate associated with the Re concentration captures only the within-sample variability. Therefore, as discussed above, this method probably underestimates the uncertainty in Re concentration for that section.

Although the uncertainty associated with the Re concentration for the CRB sections in FS-38B is calculated and reported, the estimate is likely to be lower than the actual levels that will be obtained even when more samples are taken from each section. This would be the case if the effective reduction in the uncertainty estimates achieved by collecting additional samples is not enough to compensate for the increase in the uncertainty estimates due to including between-sample variability.

4.0 Analysis of CRB

This section describes the analysis process. FS-38A was not spiked with Re and samples from this test were used to gather additional information about the background extractable Re levels in the CRB and feed materials used for FS tests. FS-38B was spiked with Re and supplied preliminary information about Re deposition in FS refractories.

4.1 Experimental Materials and Analysis Methods

As discussed, each CRB sample was broken into two segments for analysis. Each segment of dry refractory material was weighed, placed into a known volume of 0.001 M nitric acid (HNO_3), and vacuum saturated at a pressure of 84.6 kPa (12.3 lb/in.²) for no less than four hours. After saturating each sample, an aliquot of the saturation solution was removed for analysis. The sample segment was lifted out of the saturation solution and allowed to drip dry so there was no free liquid. In the second step in the extraction, the refractory segment was spun between 2000 and 5000 revolutions per minute (rpm) in a centrifuge for one hour to release non-gravity drainable pore liquid. A sample of the centrifuge-extracted liquid was collected for each segment. The two-part extraction process (saturation and centrifugation) was repeated until the majority of the Re was leached from each sample, typically accomplished with five consecutive two-part extractions. The solution composition from each extract was determined by analyzing for Re via inductively-coupled plasma-mass spectrometry.

4.2 Estimation of Background Extractable Re Concentration

The extractable Re background level in the CRB has been an ongoing question. Table 1 shows extraction data from the various CRB blank samples that have been analyzed. Early analyses of an untreated refractory blank sample showed that the extractable Re background was very low and represented less than 0.02% of the lowest Re spike used in any of the ES tests if the background was uniformly distributed over a 300-kg ES CRB. This low blank level indicated that background corrections would not be required. However, four additional samples from an ES starter-path test that contained no Re spike showed background values that varied by more than two orders of magnitude and had an average value that, if uniformly distributed over a 300-kg ES CRB, would represent 2% of the Re spiked into ES-31B, a level comparable to total Re values in the ES-31B CRB. The discrepancy in the extractable Re backgrounds of the untreated material and the starter-path blanks was attributed to residual Re in the equipment used for previous Re-spiked ES bulk vitrification tests or starter-path materials. Based on the Re results from the untreated refractory and the potential uncertainty associated with the starter-path samples, a conservative approach (in the sense that all Re in the CRB was attributed to migration from the glass melt) was adopted, and no background corrections were made to the data presented in Pierce et al. (2006).

The CRB from FS-38A presented an opportunity to gain additional information on extractable Re background levels at FS because no Re spike was added. Two FS-38A samples were taken from opposite CRB side walls across from the electrodes, midway between the floor of the box and the final level of the glass melt. The thermal environment of the samples would be similar. These two samples had extractable Re background levels that differed by an order of magnitude. Each was analyzed as two segments, and each segment showed some variation, but not as much as the sample-to-sample variations.

Table 1. Refractory Blank Concentrations

Description	Sample ID	Re Concentration ($\mu\text{g Re/kg of CRB}$) ^(a)	
		Blank Level	Error
Untreated Refractory	Refractory Blank	0.17	± 0.01
Heat-Treated Manufactured Blanks	MF-BLK-01	1.04	± 0.05
	MF-BLK-02	0.98	± 0.05
Engineering-Scale Blanks from Starter- Path Tests	EST-Blank-1	0.17	± 0.01
	EST-Blank-2	8.12	± 0.32
	EST-Blank-3	24.35	± 0.92
	EST-Blank-4	41.88	± 1.60
	Ave EST Blanks	18.63	± 0.71
FS-38A Full-Scale Blanks	FS_38A_01 Inner Segment	18.79	± 0.75
	FS_38A_01 Outer Segment	5.72	± 0.26
	FS_38A_01 Sample Average	12.11	± 0.39
	FS_38A_02 Inner Segment	0.58	± 0.04
	FS_38A_02 Outer Segment	1.78	± 0.09
	FS_38A_02 Sample Average	1.08	± 0.04
(a) Re Concentration refers to the concentration of Re in the CRB so the units $\mu\text{g/kg}$ mean μg of Re per kg of CRB. For simplicity, subsequent references to the units of Re concentration in the CRB will also just use $\mu\text{g/kg}$.			

Additional tests were conducted to verify that the source of the background was the CRB material itself and to confirm that the background was not a sampling/analytical chemistry carryover concern. Two cast CRB samples were obtained from the CRB manufacturer and heat treated at 1100°C for 8 hours to simulate the heat treatment that the CRB would see during a bulk vitrification test. The manufactured blanks were heated in a muffle arrangement to ensure that Re contamination from earlier furnace loads would not contribute to the background of these samples. The two manufactured blanks showed fairly consistent results with Re concentrations similar to that seen in the lower background FS-38A sample. This result indicates that the background level is in the CRB material.

The results from the sampling/analytical chemistry carryover tests (Table 2) show that Re was not present in a blank of the extraction solution or in either of the two cleaned containers that were run with no CRB samples with solution concentrations below the detection limit (<0.05 ppb). In contrast, solution concentrations from the first extraction from both the manufactured blanks and the FS-38A full-scale blanks were 100 to 1000 times greater than the detection limit, giving a clear indication that the extractable Re backgrounds are real and not a sample preparation or analytical chemistry concern.

Based on the background results, it appears that a soluble Re background level exists in the CRB material. The background level is highly variable and ranges from levels that are low enough that background corrections can be ignored to levels that will significantly affect the results of the Re tests. Heating the CRB appears to result in higher background levels. The cause of higher levels after heating has not been determined but may be the result of CRB porosity coarsening that allows more of the CRB

Table 2. Refractory Blank Solution Concentration

Sample Description	Sample ID	Re (ppb)
Starting extract solution used for FS-38A and FS-38B samples ^(a)	Starting solution	<0.05
Extraction of cleaned containers to determine if there was possible contaminant carryover	EXP-BLK-1-2	<0.05
	EXP-BLK-2-2	<0.05
First centrifuge extraction solution from heat-treated manufactured blanks	MF-BLK-1-2	9.47
	MF-BLK-2-2	8.47
First centrifuge extraction solution from FS-38A samples	FS-38A-01a-2	75.0
	FS-38A-02a-2	3.16
(a) Extraction solution blanks are generally run with each group of samples.		

to be accessed by extraction liquids or volatilization/condensation reactions that increase the soluble level of Re present after heating. The variable blank will be handled in this analysis by looking at the range of background levels and determining the impacts on estimations of the Re fraction in FS-38B and scale-up factors with different background correction levels.

4.3 FS-38B Soluble Re Fraction Results

The extracted Re masses from the inner and outer portions of each sample were summed separately and divided by the refractory mass to determine the Re concentration in each portion. The mass weighted average value of the inner and outer portions was computed to determine the average value for each sample. The results without any background corrections are shown in Table 3 in units of micrograms of Re per kilogram of refractory (i.e., ppb mass). The results suggest that Re is being concentrated at the melt line, which may be associated with the large buildup of sulfate salt that was observed in this region of the refractory. The wall samples show a general trend of higher Re concentrations in the higher samples, which is similar to that in several of the ES scale tests. The incomplete melt in FS-38B allows us to see and examine the Re penetration into the upper wall sections where normally the fill is only 5/8 complete. These upper wall sections (1 and 2) show relatively low Re penetration, indicating that molten salt penetration is the main Re migration path, while the volatilization/condensation onto the upper wall is less important. (See Bagaasen et al. 2006 for further discussion of Re and ⁹⁹Tc penetration mechanisms.)

The results also show that the Re penetrates deeper into the CRB wall than that noted in the ES tests, where Re concentrations fell significantly at depths greater than 1 inch. In fact, all of the samples except those taken from section 3 and one of the base samples show higher Re levels in the outer section of the CRB. This result will need to be explored further in FS-38C. The deeper penetration indicates that the Re content of sand samples from FS-38C should be evaluated to see if Re penetration ends in the CRB or continues into the sand layer. A possible reason for this observed difference is the longer duration of the FS tests, which results in higher CRB temperatures. These higher temperatures may allow vaporization/condensation reactions to drive Re penetration deeper into the CRB. The difference in the salt penetration may also be related to the nonprototypic dry waste simulant feed that is used in the FS tests in contrast to the wet simulant preparation method that was used to simulate the drying operations in the Demonstration Bulk Vitrification System for ES waste feeds. The dry waste simulant is not fully mixed with the soil and

Table 3. Re Distribution for FS-38B

Section Description	Sample ID	Re Concentration ($\mu\text{g Re/kg CRB}$)			
		Inner Sample	Outer Sample	Sample Average	Error
Section 1, vertical top	FS-38B-01	5	21	13.3	± 0.4
	FS-38B-02	5	38	21.0	± 0.7
Section 2, above melt line	FS-38B-03	24	54	39.3	± 1.8
	FS-38B-04	14	29	22.3	± 0.8
Section 3, at melt line	FS-38B-05	1197	14	571.8	± 36.9
	FS-38B-06	989	73	573.1	± 45.7
Section 4, below melt line	FS-38B-07	49	516	233.5	± 9.6
	FS-38B-08	33	105	58.7	± 1.9
Section 5, lower wall	FS-38B-11	NA ^(a)	NA	27.7	± 1.2
Section 6, refractory base	FS-38B-09 ^(b)	1	221	80.8	± 3.5
	FS-38B-10 ^(c)	3	2	2.2	± 0.1

(a) NA = not available for this sample.
(b) South electrode.
(c) North electrode.

consists of individual chemical species, some of which melt at very low temperatures.^(a) This dry simulant may allow more salt migration than waste simulant that is fully mixed with the soil in a mixer dryer operation.

The duplicate wall samples taken from wall sections 1–4 show that the Re concentration levels from two different samples from the same area are fairly consistent. The results also indicate that the analysis method for leachable Re from a CRB gives more reproducible results for samples with higher Re concentrations, although some of the variation seen in the lower concentration samples may be actual sample-to-sample variation.

The north base sample (FS-38B-10) showed low Re levels similar to those seen in ES tests. The south base sample (FS-38B-09) showed a relatively high Re level in the outer half of the sample. The increased Re concentration may be the result of molten salt penetrating along a fracture or joint in the refractory. This potential cause is supported by the qualitative observation of a dark stain in the south electrode base sample shown in Figure 9. A similar stain is seen in the north electrode area but not in the area where the extraction sample was taken. The original samples taken from the base were not the correct configuration for extraction tests, and the second set of FS-38B base samples that were collected two months later had been exposed to the weather. The impact of precipitation is expected to be minimal because it is not easy to extract water that has penetrated the CRB, but it is possible that Re may have been concentrated in different areas or leached from the base samples.

(a) For example, NaNO_2 melts at 271°C .

5.0 Calculations

The calculations described in this report are intended to provide a preliminary estimate of the amount of Re deposited in or on the CRB of FS-38B, a FS nonradioactive test of the bulk vitrification process. In-depth descriptions of the calculation methods and equations are provided in the appendix. This section describes the results of the calculations obtained by applying the methods and equations from the appendix to the FS-38B test data (see Section 4.3).

5.1 Definition of Calculated Parameters

The quantities of primary interest that were calculated relative to the CRB for FS-38B were estimates of the total extractable Re mass (g), the Re fraction (unitless), and the Re scale-up factor (unitless). Uncertainties associated with these quantities were also estimated. The Re fraction in the CRB is defined as the fraction of Re spiked into the bulk vitrification feed that deposits in or on the CRB and is present in a soluble form. The Re scale-up factor is defined as the ratio of Re fraction for ES tests to the Re fraction for FS tests. The calculations used to estimate the quantities of interest involve nominal or estimated values for several key input variables: refractory volume, specific extractable Re mass (concentration), refractory density, and Re inventory (Re spike). Estimating the uncertainties associated with the total extractable Re mass, the Re fraction, and the Re scale-up factor also involves the input variables refractory volume, specific extractable Re mass, refractory density, and Re inventory as well as estimates of the uncertainties associated with each of these input variables.

Volume approximations by vertical section (described in Section 3.1) are used, along with specific extractable Re masses for each of the sections and the overall estimate of the refractory density, to calculate estimates of extractable Re mass (also referred to as soluble Re mass) by section. Summing the sectional estimates of extractable Re mass over the different sections of the refractory then provides an estimate of the total extractable Re mass for the CRB. Dividing this total by the total Re inventory for FS-38B produces the Re fraction estimate for the CRB of FS-38B. The Re scale-up factor for FS-38B is determined by dividing an estimate of the Re fraction for ES tests by the Re fraction for FS-38B. The estimate of Re fraction for ES tests is based on calculations conducted using data from past ES tests of bulk vitrification systems. The value used to represent Re fraction for ES tests before background correction was 0.0211 (Pierce et al. 2006). This value is the mean Re fraction value over six ES tests. Table A.1 of the appendix contains the Re fraction data from the ES tests used to calculate this mean value. Methods for background corrections are discussed in Section 5.4.

Error propagation methods were used to estimate the uncertainty associated with the Re fraction and the Re scale-up factor but were not needed for estimating the uncertainty associated with the total extractable Re mass in the CRB. See discussion of error propagation and Equations (A.10), (A.12), (A.14), and (A.16) given in the appendix for further details.

5.2 Values and Uncertainties for Input Variables

Values for the input variables of volume and Re concentration were approximated for each vertical section of the CRB. Measured values for refractory density and Re inventory applied to the entire CRB.

The value used for CRB density was 2611 g/L (or 163 lb/ft³); the value used for the overall Re inventory was 229.52 g. Nominal values of 5% and 10% were used to represent the relative uncertainties associated with the CRB density and Re inventory input variables, respectively. These nominal values were considered to be reasonable for FS-38B. The uncertainty associated with the volume approximations had a potentially greater impact on the uncertainty in the quantities of interest (total extractable Re mass, the Re fraction, and the Re scale-up factor). Therefore, several values (relative values of 5, 10, 20, and 30%) representing this uncertainty were considered, covering what was considered an adequate range of potential values. Calculated values were used to represent the uncertainty associated with the input variable specific extractable Re mass for each CRB section. Additional details concerning the input variables and corresponding equations are provided in the appendix.

5.3 Penetration Depth Assumptions

Engineering-scale tests suggested that Re deposition would only be significant within the first few inches of the CRB and that little, if any, Re would penetrate deeper from the glass interface into the CRB (Pierce et al. 2006). However, such was not the case for FS-38B. When samples from FS-38B were analyzed, they were typically divided into inner and outer portions. The inner portion is the fragment that was in contact with the glass surface; the outer portion is the fragment that was away from the glass surface along the outside boundary of the CRB (see Figure 6). For most samples, Re concentrations were actually higher for sample fragments from the outer portion of the sample than from the inner portion. A paired t-test was conducted to determine whether there was a significant difference in Re concentrations between the inner and outer portions of samples from FS-38B. The t-test found that, while Re concentrations were typically higher for the outer portions of samples, these differences were not significant. Still, the sample data indicate that Re deposition was not greater for inner fragments than for outer fragments. Therefore, based on the samples available from FS-38B, it was assumed for calculational purposes that Re penetration affected at least the entire 6-inch CRB wall thickness. Because only a few samples were taken from the bottom wall section and the CRB base, it was not possible to determine whether Re penetration beyond 6 inches was significant. Therefore, calculations were conducted based on both the full CRB volume (assuming the entire CRB was significantly affected by Re penetration), and on the penetrated volume corresponding to a penetration depth of 6 inches, the assumed thickness of the CRB walls. It is important to consider both cases because the estimate of extractable Re in the CRB is a function of affected volume. If the entire CRB volume was in fact affected by Re penetration, the approximation of full volume should be used when calculating quantities of interest that describe Re deposition. However, if only part of the CRB was actually affected by Re penetration, using the full volume approximation when calculating quantities of interest would result in an overestimate of extractable Re mass in the CRB, and an approximate penetrated volume should be used instead. For calculations based on penetrated volume, a nominal relative uncertainty of 10% was used to represent the uncertainty associated with the penetration depth. Additional details concerning volume uncertainties are provided in the appendix.

5.4 Treatment of CRB Extractable Re Background Levels

Section 4.2 discusses the background levels of extractable Re measured in various CRB blank samples. The background level is highly variable and ranges from levels that are low enough that background corrections can be ignored to levels that will significantly affect the results of the Re tests. If background level adjustments are made in the FS-38B data, it will also be necessary to make similar

adjustments to ES test results in order to not bias the Re scale-up factor because no previous background corrections were made for these test results.

Several estimates of Re fraction for both ES tests and test FS-38B were used in calculating the Re scale-up factor. These estimates of Re fraction are based on applying several different background adjustment quantities to the Re concentration data from the ES tests and from test FS-38B. Table 1 provides information on background extractable Re found in blank CRB samples from various ES and FS tests. The adjustment quantities used in the calculations presented in this report were 0, 1, 10, 20, 30, and 40 µg/kg. These quantities represent the range of observed values contained in Table 1. The adjustments for background extractable Re were made by subtracting the specified adjustment quantities from the analyzed Re concentrations given in Table 3 for test FS-38B and from analyzed Re concentrations obtained from past ES tests. If the subtraction resulted in a negative value, a 0 concentration was used for that section. In calculating estimates of the Re scale-up factor, the same adjustment amount was applied to the Re fraction estimates from both the ES tests and test FS-38B. The estimates of Re fraction for test FS-38B involve the above mentioned background adjustment quantities and are summarized in Table A.1 of the appendix. The estimates of Re fraction for ES tests are based on calculations conducted using data from past ES tests of bulk vitrification systems and involve the same adjustment quantities. Table A.2 of Appendix A provides details of the Re fraction calculations for the ES tests.

5.5 Assumptions Associated with Calculations

The calculations conducted to describe Re deposition in the CRB of FS-38B are based on several underlying assumptions. First, it is assumed that the vertical sections defined previously are adequate for describing the vertical effect in Re deposition in the CRB of FS-38B. Second, it is assumed that Re deposition is homogeneous within each vertical section, and that the estimates of Re concentration obtained by analyzing CRB samples accurately describe Re deposition within each section. Statistical sampling plans are intended to ensure, to the extent possible, that such assumptions are valid. The limited samples ultimately available for FS-38B were not sufficient to validate these assumptions. Still, it was thought that analyses based on the available CRB samples for FS-38B would provide at least some preliminary information about Re deposition in FS tests.

5.6 Calculation Results

The results of calculations are presented in Tables 4 through 10. The first section of the calculation results describes how the Re is distributed in sections of the FS CRB (Section 5.6.1). Section 5.6.2 examines the impacts of different assumed affected volumes and the volume uncertainty estimates. Section 5.6.3 describes the impacts of different extractable Re background levels. Section 5.6.4 gives a range of estimates for extractable ⁹⁹Tc levels in a FS CRB.

5.6.1 Re Distribution in Different Sections of the FS CRB

Table 4 lists the approximate CRB volume proportions by vertical section and the relative amount of Re in each section. Data in this table show that section 3, at the glass melt line, contains about 50% of the total extractable Re even though it is the smallest of the CRB sections. Section 4, located just below the

melt line, also was a small fraction of the total refractory volume but contained about 20% of the extractable Re. The upper two sections (sections 1 and 2) of the CRB represented 30.4 to 37.8% of the volume but less than 9% of the extractable Re. The lower wall, section 5, showed low extractable Re quantities. This distribution in the CRB wall roughly supports a model that has less extractable Re in the lower portions of the CRB due to smaller quantities of molten salt and/or better incorporation of Re into an insoluble phase as the CRB temperature increases. Higher extractable Re values are found near the final glass surface where nitrate salts from newly added feed can combine with sulfate salts that build up at the glass surface and penetrate into the CRB. The region above the CRB shows some contamination due to volatilization/ condensation reactions, but not as high as the lower sections because no molten salt penetration occurs. However, the support is not statistically significant because the wall samples were taken as duplicates from only one location and the variation of extractable Re at a given height at different locations around the box was not determined.

The base section of the FS CRB is a significant fraction of the overall CRB volume. The extractable Re in this section is relatively high, but this is based largely on the high Re concentration seen in the outer section of one of base samples. Table 4 also shows that the main difference in the 6-inch penetrated volume case and the full volume case is in the affected portion of the base section. Using a 6-inch penetration depth reduces the relative amount of material in the base from 34.7 to 24.6% and the relative amount of Re in the base from 17.2 to 10.8%. Had the penetration levels into the base of the FS CRB been similar to the ES CRB, a 6-inch or less penetration depth would have been used to eliminate portions of the base that were not penetrated by Re. However, the Re concentrations measured in the one outer section of the base indicated that the more conservative assumption of full volume penetration should be used for the FS-38B CRB.

Table 4. Approximate Volume and Re Percentages by CRB Section

Volume Approximation Method	Section Volume (% of total penetrated volume)					
	1	2	3	4	5	6
Full volume	21.2	9.2	7.3	11.4	16.2	34.7
6-inch penetrated volume	26.4	11.4	9.1	14.2	14.3	24.6
	Re Quantity by Section (% of total Re present in CRB)					
Full volume	4.4	3.4	49.8	19.9	5.4	17.2
6-inch penetrated volume	4.8	3.7	54.7	21.8	4.2	10.8

5.6.2 Impacts of Affected Volumes and Volume Uncertainty Estimates

Table 5 shows total extractable Re, the Re fraction, and the Re scale-up factor determined when no background correction is made under various assumptions relating to affected volumes and volume uncertainty estimates. The estimated total extractable Re ranges from 1.57 to 1.72 g, leading to Re fractions in the CRB that range from 0.0068 to 0.0075, or 0.68 to 0.75% of the 229.52 g of Re that were spiked into the FS-38B test. This Re fraction results in ES/FS scale-up factors that range from 2.81 to 3.09.

Table 5. Results Based on Full Volume and 6-inch Penetrated Volume Approximations Without Background Corrections

Relative Volume Uncertainty	Total Re (g)	SD Total Re (g)	Re Fraction	SD of Re Fraction	Relative Uncertainty for Re Fraction	Re Scale-up Factor	SD of Re Scale-up Factor	Relative Uncertainty for Re Scale-up Factor
Full Volume Approximations								
5%	1.72	0.36	0.0075	0.0017	0.23	2.81	1.19	0.42
10%	1.72	0.37	0.0075	0.0018	0.24	2.81	1.19	0.43
20%	1.72	0.41	0.0075	0.0019	0.26	2.81	1.23	0.44
30%	1.72	0.46	0.0075	0.0022	0.29	2.81	1.28	0.45
6-inch Penetrated Volume Approximations								
5%	1.57	0.28	0.0068	0.0014	0.20	3.09	1.26	0.41
10%	1.57	0.29	0.0068	0.0014	0.21	3.09	1.27	0.41
20%	1.57	0.33	0.0068	0.0016	0.23	3.09	1.31	0.42
30%	1.57	0.39	0.0068	0.0018	0.27	3.09	1.37	0.44

The results in Table 5 indicate that, for the FS CRB, there is less than 10% difference between the total extractable Re, the Re fraction, and the Re scale-up factor calculated assuming a penetration depth of 6 inches and using the full volume of the refractory. As discussed previously, the levels of Re in the outer sections of the FS CRB samples dictate that the most conservative assumption, that the full volume of the CRB is penetrated by Re, is assumed in subsequent calculations. However, these results show that this conservative assumption impacts the final results of the analysis by less than 10%.

Table 5 also shows the impacts of different relative volume uncertainties on the overall uncertainty of the Re fraction and the Re scale-up factor. A relative volume uncertainty of 10 to 20% is reasonable based on the rough error propagation estimates conducted relative to estimating the CRB volume and corresponding uncertainty (see Section A.2.5 of the appendix). This range of nominal relative volume uncertainty values resulted in minimal differences in the uncertainty associated with the final Re fraction and scale-up factor estimates. More specifically, when nominal volume uncertainties of 5, 10, 20, or 30% were used as estimates of volume uncertainty, the corresponding estimated relative uncertainties in the Re fraction estimate ranged from 23 to 29% for full-volume case (assuming no adjustment in Re concentrations due to background extractable Re in blank samples) and from 20 to 27% for the 6-inch penetrated volume cases. The relative uncertainties in the Re scale-up factor estimate ranged from 42 to 45% for full-volume cases and from 41 to 44% for the 6-inch penetrated volume cases. Subsequent calculations assume a 10% relative volume uncertainty.

5.6.3 Impacts of Different Background Corrections

Section 5.4 describes the different extractable Re background levels that were evaluated. The results in Tables 6–8 show the impact of different background correction levels assuming the entire refractory was affected and a 10% volume uncertainty. The quantities listed in the tables were calculated for the zero-background case as well as for background levels ranging from a low of 1 µg/kg based on background levels in the low FS-38A LS blank to 40 µg/kg based on the highest backgrounds seen in starter-

path ES CRB samples. Background levels were subtracted from each section of the FS-38B CRB. If the background level exceeded the measured levels, that section was assigned to have a 0 extractable Re concentration.

Table 6 shows how different background adjustment levels affect the relative distribution of Re in the FS-38B CRB. As the background adjustment level increases, the relative amount of Re increases at and just below the final glass surface. This result indicates that efforts to reduce Re and ⁹⁹Tc migration reduction should focus on mechanisms related to molten salt migration.

Table 6. Percentage of Extractable Re Mass by CRB Section Based on Full Volume Approximations as a Function of Background Level

Extractable Re Background Adjustment Level (µg/kg)	Re Quantity by Section (% of Total Re Present in CRB)					
	1	2	3	4	5	6
0	3.80	3.43	50.18	19.97	5.41	17.21
1	3.59	3.36	50.70	20.08	5.27	17.00
10	1.82	2.59	54.81	20.68	3.84	16.27
20	0.44	1.50	59.57	21.20	1.85	15.44
30	0.00	0.71	63.91	21.32	0.00	14.07
40	0.00	0.00	67.11	20.84	0.00	12.05

Table 7 shows how different background adjustment levels affect total extractable Re and Re fraction. As expected, the higher background correction levels reduce the extractable Re and Re fractions attributable to migration from the waste feed. However, even the highest measured background levels drop the values by only 30%, so extractable Re that originates from the waste feed clearly exists in the FS CRB.

Table 7. Results Based on Full-Volume Approximations and 10% Volume Uncertainty

Extractable Re Background Adjustment Level (µg/kg)	Total Re (g)	SD Total Re (g)	Re Fraction	SD of Re Fraction	Relative Uncertainty for Re Fraction	Re Scale-up Factor	SD of Re Scale-up Factor	Relative Uncertainty for Re Scale-up Factor
0	1.71	0.36	0.0075	0.0018	0.23	2.83	1.20	0.42
1	1.69	0.36	0.0074	0.0017	0.24	2.82	1.21	0.43
10	1.54	0.34	0.0067	0.0016	0.24	2.72	1.30	0.48
20	1.39	0.31	0.0061	0.0015	0.25	2.72	1.37	0.50
30	1.28	0.29 ^(a)	0.0056	0.0014 ^(a)	0.25 ^(a)	2.71	1.40 ^(a)	0.52 ^(a)
40	1.19	0.27 ^(a)	0.0052	0.0013 ^(a)	0.25 ^(a)	2.65	1.41 ^(a)	0.53 ^(a)

(a) These values were calculated using only data from CRB sections having Re concentrations above the specified background levels.

Table 7 also shows that the Re scale-up factor is fairly independent of the background adjustment and drops by about 6% at the highest adjustment. Table 8 shows that the drop in the Re scale-up factor is due to a background correction that has a slightly greater effect on ES-scale test results than FS results. This indicates that, although the uncertainty associated with extractable Re background levels adds significant uncertainty to the actual levels of extractable Re attributable to migration from the waste feed, different background level assumptions do not significantly change the scale-up factor estimated from this test.

Table 8. Re Fraction and Scale-up Values for Different Background Adjustment Amounts Based on Full-Volume Approximations

Re Concentration Adjustment Amount ($\mu\text{g}/\text{kg}$)	0	1	10	20	30	40
ES Re Fraction	0.021	0.021	0.018	0.016	0.015	0.014
FS Re Fraction	0.0075	0.0074	0.0067	0.0061	0.0056	0.0052
Re Scale-up Factor	2.81	2.80	2.72	2.72	2.70	2.64

Table 9 shows the effect of different extractable Re background levels on the $^{99}\text{Tc}/\text{Re}$ mobility ratio that is determined from ES-32A and ES-32B that contained both Re and ^{99}Tc spikes.^(a) The table shows the ratio for each section of the CRB and the average determined from the section values for different background correction levels. This is the method that was used in Pierce et al. (2006). The background correction levels do not significantly affect the ratio in ES-32A (values vary from 0.22 to 0.24). However, the results from ES-32B are significantly impacted for a background correction of 10 $\mu\text{g}/\text{kg}$ or greater because the measured level of Re is less than the blank level in one or more sections, and the denominator in the $^{99}\text{Tc}/\text{Re}$ mobility ratio goes to 0. The background level has a greater impact in ES-32B because the Re spike level is only 10% of that used for ES-32A. The lower spike level means that a given fraction of Re in the CRB results in a concentration that is 10 times lower in ES-32B. For example, if a 1% Re migration into the refractory resulted in a concentration of X for ES-32B, the same fraction of Re migration in ES-32A would result in a concentration of 10X. Subtracting a background of X has a significant effect on ES-32B results but little effect on ES-32A results. The lower spike level in ES-32B was intended to better match the expected ^{99}Tc concentrations that would be present, but it also had the unexpected consequence of being more sensitive to extractable Re background levels. Table 9 also shows $^{99}\text{Tc}/\text{Re}$ mobility ratios that are calculated based on the ratio of ^{99}Tc and Re mass fractions for the entire CRB. This method gives similar results for ES-32A but avoids the 0 concentrations for ES-32B. Using data from both tests in combination give $^{99}\text{Tc}/\text{Re}$ mobility ratios ranging from 0.17 to 0.41.

(a) ^{99}Tc background concentrations in CRB blank samples are less than detection limits (Pierce 2005), so background corrections are only necessary for Re.

Table 9. ⁹⁹Tc/Re Mobility Ratios for Different Background Adjustment Amounts

Re Concentration Adjustment Amount (µg/kg)	⁹⁹ Tc/Re Mobility Ratios					
	0	1	10	20	30	40
ES-32A with Re Spike Level of 7.51 g						
Section 1	0.14	0.14	0.14	0.15	0.15	0.15
Section 2	0.21	0.21	0.21	0.22	0.23	0.23
Section 3	0.42	0.42	0.43	0.44	0.45	0.47
Section 4	0.94	0.95	1.04	1.16	1.32	1.53
Section 5	0.11	0.11	0.12	0.12	0.12	0.12
Sum of All Sections ^(a)	0.24	0.24	0.24	0.25	0.26	0.27
Average of Sections 1-3 and 5 ^(a,b)	0.22	0.22	0.23	0.23	0.24	0.24
ES-32B with Re Spike Level of 0.709 g						
Section 1	0.17	0.17	0.19	0.22	0.27	0.32
Section 2	0.15	0.15	0.21	0.37	1.53	NC ^(c)
Section 3	0.16	0.18	1.76	NC	NC	NC
Section 4	0.08	0.09	NC	NC	NC	NC
Section 5	0.00	0.01	NC	NC	NC	NC
Sum of All Sections ^(a)	0.15	0.16	0.23	0.30	0.41	0.54
Average of Sections 1–5 ^(a)	0.11	0.12	NC	NC	NC	NC
ES-32A and ES-32B Combined Results						
Average of Sums of All Sections	0.19	0.20	0.24	0.27	0.34	0.41
Average of Section Averages	0.17	0.17	NC	NC	NC	NC
(a) Sum of all sections is the ratio of ⁹⁹ Tc and Re mass fractions for the entire CRB. The average of sections first calculates the ratio of ⁹⁹ Tc and Re mass fractions for each section and then takes the average.						
(b) Section 4 was not included in Pierce et al. (2006) because it was thought to be an outlier.						
(c) NC indicates that the value was not calculated for this background correction level because the measured level of Re is less than the blank level, resulting in a denominator of zero.						

5.6.4 Projected ⁹⁹Tc Levels in FS CRB

Using an approach similar to McGrail et al. (2003), the Re information collected from FS-38B and the ⁹⁹Tc data collected at ES were used to project the amount of ⁹⁹Tc that might be present in a FS CRB. This methodology uses a ⁹⁹Tc/Re mobility ratio determined from ES tests to adjust the Re levels measured in FS tests. Multiplying the Re levels determined from FS-38B by the ⁹⁹Tc/Re mobility ratio supplies an estimate of the anticipated ⁹⁹Tc concentrations that might be present at full scale. Two ES tests that were spiked with both Tc and Re showed that Re was more mobile with an average ⁹⁹Tc/Re mobility ratio of 0.17 ± 0.12 when no correction is made for extractable background Re (Pierce et al. 2006). The data in Section 5.6.3 showed that accounting for different possible extractable Re background values leads to ⁹⁹Tc/Re mobility ratios that vary from 0.17 to 0.41 (see Table 9). When these values were used with FS-38B Re fractions, the projected FS ⁹⁹Tc fraction ranged from 0.0013 to 0.0021, or 0.13 to 0.21% for extractable Re background corrections that ranged from 0 to 40 µg/kg (see Table 10).

Table 10. FS CRB ⁹⁹Tc Fractions Projected from FS-38B Re Results

Re Concentration Adjustment (µg/kg)	0^(a)	0^(b)	1	10	20	30	40
FS-38B CRB Re fraction	0.0075	0.0075	0.0074	0.0067	0.0061	0.0056	0.0052
⁹⁹ Tc/Re mobility ratio	0.17 ^(a)	0.19 ^(b)	0.20 ^(b)	0.24 ^(b)	0.27 ^(b)	0.34 ^(b)	0.41 ^(b)
Projected FS CRB ⁹⁹ Tc fraction	0.0013	0.0015	0.0015	0.0016	0.0017	0.0019	0.0021
(a) Based on ⁹⁹ Tc/Re mobility ratio calculated by averaging the total of ratios determined for each section.							
(b) Based on ⁹⁹ Tc/Re mobility ratio calculated by determining the ⁹⁹ Tc and Re mass fractions for the entire CRB and calculating a single ratio.							

6.0 Conclusions/Recommendations

Although the FS-38B tests did not generate a full box of glass, the abbreviated sampling plan implemented was useful for two main reasons. First, the analysis of these FS samples clarified the sampling and analysis techniques needed to obtain the required information from the full analysis planned in test FS-38C. Second, these limited analyses pointed out several differences between ES and FS test results and thus were very useful in advancing the understanding of Re and ⁹⁹Tc migration into the CRB at full scale. The key conclusions and recommendations from the FS-38B analyses of the CRB are listed below.

6.1 Conclusions

- The CRB material contains a measurable extractable Re background level. This level is highly variable, ranging from 1 µg/kg, which does not require background adjustments, to more than 40 µg/kg, which requires background adjustments to correctly interpret the Re results. The uncertainty of the extractable Re background level is dealt with in this report by reporting ranges of values for different extractable Re background adjustments.
- Estimates of the extractable Re fraction in the FS-38B CRB ranged from 0.75% with no background adjustment to 0.52% for a background adjustment of 40 µg/kg.
- The initial FS-38B results indicate that a smaller fraction of the Re present in the feed moves into the CRB at FS than at ES. The ES/FS scale-up factor was fairly independent of the background adjustment levels and ranged from 2.81 to 2.61.
- FS CRB ⁹⁹Tc fractions in the CRB projected from FS-38B Re results ranged from 0.13 to 0.21% depending on the level of extractable Re background correction.
- Careful sampling of the CRB is required to obtain usable samples. The samples must be a continuous piece that represents the entire wall thickness.
- The two segments used to analyze the extractable Re concentration in each refractory sample indicated that Re penetrates deeper into the FS CRB than in the ES CRB.
- Duplicate CRB samples from same locations showed reasonably good agreement in the concentration of extractable Re.
- The two base samples showed significantly different results, demonstrating the need to collect multiple samples from each vertical section of the CRB to assess variability of Re concentration in different parts of the box and obtain a more reliable estimate of extractable Re present.
- Although the abbreviated CRB sampling routine used for FS-38B did not allow examination of horizontal concentration effects, the limited data did indicate a considerable vertical effect with significant concentration of extractable Re in the CRB at the melt line.
- Assumptions about affected volumes and volume uncertainty did not have a significant effect on the amount of or uncertainty in extractable Re estimates.

6.2 Recommendations

- The FS CRB samples taken for Re analysis must meet specifications to be useful. It is recommended that personnel familiar with the refractory analysis process be present during CRB sampling. The CRB should be retained and protected from the environment until all samples have been transferred and approved by the analysis organization.
- The deeper penetration of Re into the FS CRB indicates that the Re levels in the sand between the CRB and the bulk vitrification container should be measured in subsequent FS tests. The extractable Re background level of the sand should also be determined as part of the sand testing.
- The analysis of the CRB samples as inner and outer segments provided useful information about the Re distribution in the FS CRB. Although combining the solutions produced for each sample would reduce the number of ICP-MS samples by half, it is recommended that these solutions continue to be analyzed separately to monitor penetration levels.
- Future FS tests should continue to investigate spatial effects on Re deposition in the CRB. The potential for significant vertical and horizontal effects should be explored.
- The uncertainty associated with the extractable Re background level should be minimized by analyzing multiple blank samples of the FS-38C CRB material.
- The variable extractable Re background indicates that any future ES tests should use larger Re spike levels to reduce the effect of CRB extractable Re background levels on the final results.

7.0 References

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Appendix
Calculations

Appendix - Calculations

As discussed in Section 5 of the main report, certain input variables were used to calculate estimates of key quantities of interest relative to rhenium (Re) deposition in the castable refractory block (CRB) during full-scale (FS) test FS-38B. The input variables needed to conduct the calculations are CRB volume approximations, specific extractable Re mass, refractory density, and Re inventory. The calculated quantities of interest are total extractable Re, Re fraction, and the Re scale-up factor. These input variables and calculated quantities, together with their respective uncertainties, are discussed in this appendix. Error propagation was used to approximate uncertainty associated with some calculated quantities of interest. Thus, a brief explanation of applicable error propagation formulas is also presented.

A.2 Input Variables

A.2.1 Volume Approximations

The refractory volume (in liters) is an approximated input variable. Because the vertical sections all have a somewhat different shape (including wall sections and the base), volume must be approximated for each section. Furthermore, the volumes discussed here and used for subsequent calculations include both actual volumes and the volume of each section penetrated by Re. Section 5.3 of the main report discussed the need to conduct calculations based on both approximate full volumes and approximate penetrated volumes. Because volume approximation is the most complicated part of the process leading to Re fraction and scale-up factor estimates, additional details concerning volume calculations are presented in this appendix. For calculational purposes, the uncertainties associated with the volume approximations were nominal relative amounts. Additional details concerning the uncertainties associated with the volume approximations are also given in Section A.2.5.

This section of the appendix presents the equations needed to estimate volumes for the three regions of the CRB: the upper wall, the bottom of the wall just above the base, and the base. The figures and equations presented herein are based on four upper wall sections, one bottom wall section with an angled or chamfered shape, and the base. Section 2.1 of the main report provides further details on the CRB sections. The CRB sections defined for FS-38B do not correspond to the intended sampling plan for FS-38B, which called for samples to be taken from several locations on the upper wall, at three different vertical heights, and from the CRB base. The sampling plan did not call for sampling from the bottom wall section. However, because the bottom wall section has a different shape than the upper wall sections (because of the chamfer), separate volume approximation equations are presented for that section. Future FS tests are expected to have similar CRBs, but the sampling plans could differ such that the vertical sections may be defined differently for future tests.

A.2.2 Upper CRB Wall Sections

Although the upper CRB wall actually has a slight taper, volume approximation equations for the upper wall sections are based on the assumption that the wall is exactly vertical and of uniform thickness. Figure A.1 shows a cutaway view of a portion of the CRB wall and base, with input variables used in the volume approximation equations identified. The dashed lines in Figure A.1 depict the penetration depth,

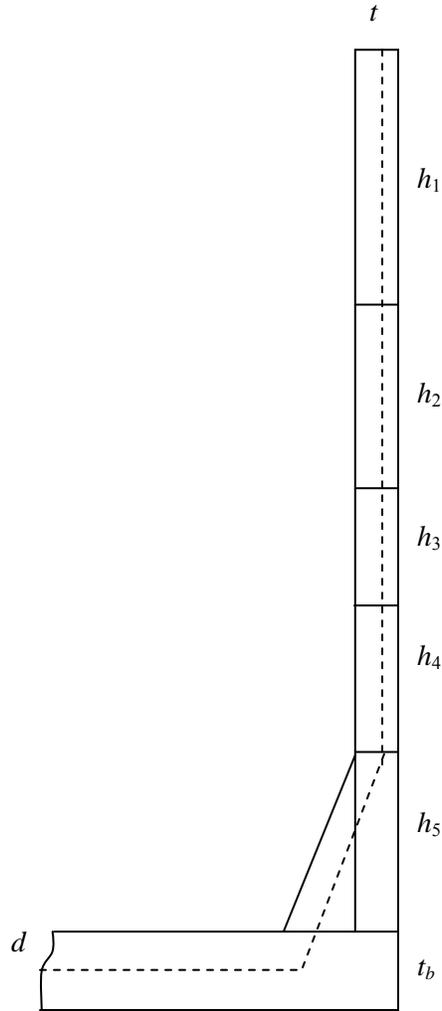


Figure A.1. Cutaway View of CRB Wall and Base with Penetration Depth Depicted

d , and horizontal lines to identify the vertical sections (including the CRB base). Heights of the vertical sections from the upper CRB wall are labeled h_1 , h_2 , h_3 , and h_4 in the figure. The height of the bottom CRB wall section is labeled h_5 , and the thickness of the CRB base section is labeled t_b , which is assumed to be 8 in. The thickness of the upper CRB wall is labeled t .

The full volume of the four upper sections of the refractory wall can be approximated by multiplying the height of each section by the area of the “donut-shaped” region of thickness t that results when the area determined by the inner surface of the CRB wall (the surface in contact with the glass) is subtracted from the area determined by the outer surface of the wall. The penetrated volume of the four upper wall sections can be approximated by multiplying the height of each section by the area of the donut-shaped region of thickness d that results when the area determined by the inner surface of the CRB wall is subtracted from the area determined by the penetration boundary within the CRB wall. These areas can be calculated by subtracting appropriate triangular regions from larger rectangular regions, as illustrated in Figure A.2.

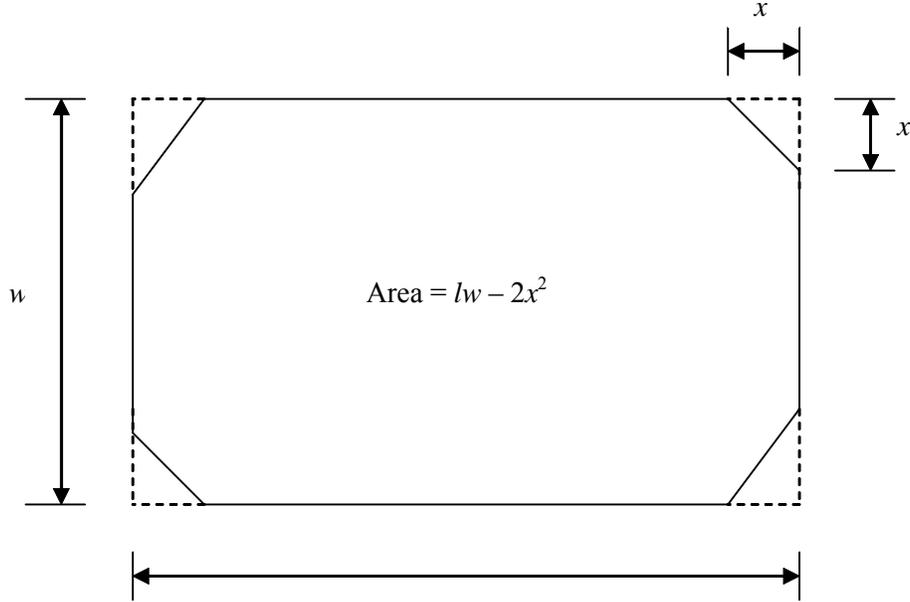


Figure A.2. Illustration of Area Calculation

The full volume of the four upper sections of the CRB wall can be approximated using the equation:

$$V_{full,i} = h_i \left[(LW - 2c^2) - (L - 2t)(W - 2t) + 2(c + t(\sqrt{2} - 2))^2 \right] \text{ for } i = 1, 2, 3, 4. \quad (\text{A.1})$$

The penetrated volume of the four upper sections of the CRB wall can be approximated by

$$V_{pen,i} = h_i \left[(L - 2(t - d))(W - 2(t - d)) - 2(c + (t - d)(\sqrt{2} - 2))^2 - (L - 2t)(W - 2t) + 2(c + t(\sqrt{2} - 2))^2 \right] \text{ for } i = 1, 2, 3, 4. \quad (\text{A.2})$$

where

- $V_{full,i}$ = approximate full volume of the i^{th} upper wall section for $i = 1, 2, 3,$ and 4
- $V_{pen,i}$ = approximate penetrated volume of the i^{th} upper wall section for $i = 1, 2, 3,$ and 4
- h_i = height of section i for $i = 1, 2, 3,$ and 4
- L = overall length of the CRB, assumed to be 23 ft
- t = thickness of the upper refractory wall in the section, assumed to be 6 in.
- d = penetration depth, can be assigned values between 0 and 6 in.
- W = overall width of the CRB, assumed to be 78 in.
- c = outer corner dimension for the overall CRB, assumed to be 18 in.

For the upper wall sections, using a penetration depth of $d = t$ in Eq. (A.2) results in Eq. (A.1). Volumes obtained using Eq. (A.1) and (A.2) are approximations because they are based on an estimate of either the CRB wall thickness or the penetration depth, on the assumption that the wall thickness or penetration depth is uniform over the four upper wall sections, and on the assumption that the refractory walls are exactly vertical. Figure A.3 is a top view of the CRB, with penetration depth depicted, and includes labels to identify L , W , and c .

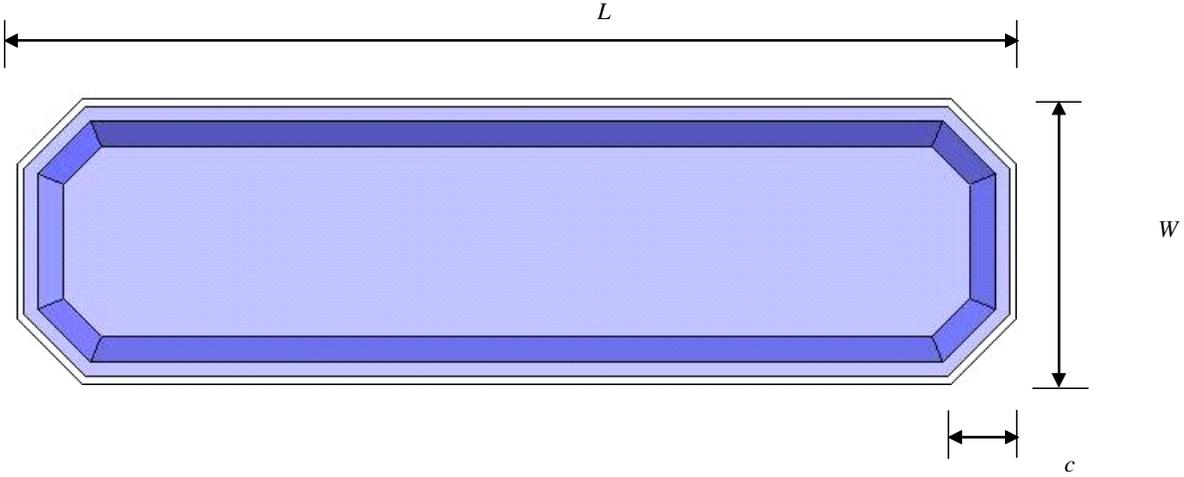


Figure A.3. Top View of CRB

A.2.3 Bottom CRB Wall Section

The bottom CRB wall section involves two parts, the region having a rectangular-shaped cross section (similar to the upper CRB wall sections) and the region having a triangular-shaped cross section. The bottom wall section is identified as section 5 in Figure A.1 and in Figure 2 in the main report. The full volume of the bottom wall section can be approximated by adding the approximated volumes of these two regions. The approximate volume of the first of these two regions can be found by applying the approach illustrated in Figure A.2. This first region is donut-shaped like the upper wall sections and thus has a volume approximation formula like Eq. (A.1) or (A.2), with h_5 used to represent the height of the region. The approximate volume of the second of these two regions can be found by subdividing the region into various pieces, including two lengths, two widths, four corner pieces, and 16 pyramid shaped pieces that lie between the corner pieces and length or widths.

Thus, the full volume of the bottom section of the refractory wall (the wall section just above the refractory base) can be approximated using the following equation:

$$V_{full,5} = h_5 \left[(LW - 2c^2) - (L - 2t)(W - 2t) + 2(c + t(\sqrt{2} - 2))^2 \right] +$$

$$2\left(\frac{1}{2}bh_5\right)(L - 2(c + (t + b)(\sqrt{2} - 1))) +$$

$$2\left(\frac{1}{2}bh_5\right)(W - 2(c + (t + b)(\sqrt{2} - 1))) +$$

$$4\sqrt{2}\left(\frac{1}{2}bh_5\right)\left(c + (t+b)(\sqrt{2}-2)\right) + 16\left(\frac{1}{3}b^2h_5(\sqrt{2}-1)\right).$$

After some simplification, the equation for approximating the full volume of the bottom CRB wall section can be written as

$$V_{full,5} = 2h_5t\left[L+W+2\left(c+t\sqrt{2}\right)\left(\sqrt{2}-2\right)\right] + bh_5\left[L+W-4\left(c+(t+b)\left(\sqrt{2}-1\right)\right) + 2\sqrt{2}\left(c+(t+b)\left(\sqrt{2}-2\right)\right) + \frac{16}{3}b\left(\sqrt{2}-1\right)\right]. \quad (\text{A.3})$$

The approximate penetrated volume of the bottom CRB wall section can be obtained by subtracting the unpenetrated part of the triangular (chamfered) region of this section from its total volume, then adding the volume of the penetrated region that lies within the vertical part of the CRB wall beyond the chamfered region. In Figure A.4, these three regions are represented by the triangles having legs (non-hypotenuse sides) labeled b and h_5 , $b-r$ and h_5-p , and r and p , respectively. The subvolumes that correspond to these three regions each involve summing volumes for two length pieces, two width pieces, four corner pieces, and 16 pyramid-shaped regions.

The penetrated volume of the bottom section of the refractory wall can be approximated using this equation:

$$\begin{aligned} V_{pen,5} = & \left[2\left(\frac{1}{2}bh_5\right)\left(L-2\left(t+b+c+(t+b)\left(\sqrt{2}-2\right)\right)\right) + \right. \\ & 2\left(\frac{1}{2}bh_5\right)\left(W-2\left(t+b+c+(t+b)\left(\sqrt{2}-2\right)\right)\right) + \\ & \left. 4\left(\frac{1}{2}bh_5\right)\left(c+(t+b)\left(\sqrt{2}-2\right)\right)\left(\sqrt{2}\right) + 16\left(\frac{1}{3}b^2h_5\left(\sqrt{2}-1\right)\right)\right] - \\ & \left[2\left(\frac{1}{2}(b-r)(h_5-p)\right)\left(L-2\left(t+b+c+(t+b)\left(\sqrt{2}-2\right)-r\left(\sqrt{2}-1\right)\right)\right) + \right. \\ & 2\left(\frac{1}{2}(b-r)(h_5-p)\right)\left(W-2\left(t+b+c+(t+b)\left(\sqrt{2}-2\right)-r\left(\sqrt{2}-1\right)\right)\right) + \\ & 4\left(\frac{1}{2}(b-r)(h_5-p)\right)\left(c+(t+b-r)\left(\sqrt{2}-2\right)\right)\left(\sqrt{2}\right) + \\ & \left. 16\left(\frac{1}{3}(b-r)^2(h_5-p)\left(\sqrt{2}-1\right)\right)\right] + \\ & \left[2\left(\frac{1}{2}pr\right)\left(L-2\left(t+c+t\left(\sqrt{2}-2\right)\right)\right) + 2\left(\frac{1}{2}pr\right)\left(W-2\left(t+c+t\left(\sqrt{2}-2\right)\right)\right) + \right. \\ & \left. 4\left(\frac{1}{2}pr\right)\left(c+t\left(\sqrt{2}-2\right)\right)\left(\sqrt{2}\right) + 16\left(\frac{1}{3}p\right)\left(\frac{1}{2}r^2\left(\sqrt{2}-1\right)\right)\right]. \end{aligned}$$

where

- $V_{full,5}$ = the approximate full volume of the bottom wall section
- $V_{pen,5}$ = the approximate penetrated volume of the bottom wall section
- b = the width of the angled portion of the bottom section of the CRB
the section just above the refractory base, assumed to be 7 in.
- h_5 = the height of bottom section of the refractory wall, assumed to be 13 in.
- r = the horizontal penetration distance beyond the angled portion of the
bottom section of the refractory wall and into the vertical portion
- p = the vertical penetration distance beyond the angled portion of the bottom section of the
refractory wall and into the vertical portion

and other quantities are as defined previously.

Figure A.4 shows a cutaway view of a portion of the CRB wall and base with labels to identify b , h_5 , r , and p . While the dashed lines in Figure A.1 represent a uniform penetration depth into the CRB walls and base, the dashed lines in Figure A.4 illustrate how the penetrated volume is actually approximated using Eq. (A.4) and (A.6). Note also that

$$r = \frac{d\sqrt{h_5^2 + b^2}}{h_5} \quad \text{and} \quad p = \frac{d\sqrt{h_5^2 + b^2}}{b}.$$

The volume $V_{full,5}$ is an approximation because it is based on the assumption that the two regions of the bottom CRB wall section are of uniform shape with the specified dimensions. The volume $V_{pen,5}$ in Eq. (A.4) is an approximation because it is based on an estimate of the penetration depth, on the assumption that the penetration depth is uniform over the angled (chamfered) portion of the bottom section and the region penetrated beyond the angled portion is accurately represented by r and p (which in turn are based on the estimate of the penetration depth, d), and on the nominal value for the width (b) of the angled portion at the bottom of the refractory wall.

A.2.4 CRB Base Section

The full volume of the CRB base (section 6) can be approximated using the approach illustrated in Figure A.2. Thus the full volume of the CRB base section can be approximated using the equation:

$$V_{full,6} = t_b(LW - 2c^2). \tag{A.5}$$

where, as depicted in Figures A.1 and A.4, t_b represents the thickness of the CRB base section, which is assumed to be 8 inches. Figure A.3 presents a description of the refractory length, width, and corner dimensions (L , W , and c , respectively).

The approximate penetrated volume of the CRB base section can be obtained by multiplying the area of the base that is not below the CRB walls (so the area that is exposed to the glass) by the penetration depth, adding the volume of the region of the base that is below the chamfered part of the bottom CRB wall section as defined by the triangle with legs r and p , then subtracting the part of that triangular region

that is past the penetration depth. The area that is exposed to the glass can be calculated following the approach illustrated in Figure A.2. The volume of the region below the chamfered part of the bottom CRB wall section is represented in Figure A.4 by the triangle with legs r and p that lies below the chamfered region and is similar to the third subvolume describing the bottom CRB wall section. The part of this triangular region that is to be subtracted is shaded in Figure A.4.

The penetrated volume of the base section of the CRB can be approximated using the equation:

$$\begin{aligned}
V_{pen,6} = & (d) \left[(L - 2(t + b))(W - 2(t + b)) - 2(c + (t + b)(\sqrt{2} - 2))^2 \right] + \\
& \left[2\left(\frac{1}{2} pr\right) \left(L - 2\left(t + b + c + (t + b)(\sqrt{2} - 2) \right) \right) + \right. \\
& \left. 2\left(\frac{1}{2} pr\right) \left(W - 2\left(t + b + c + (t + b)(\sqrt{2} - 2) \right) \right) + \right. \\
& \left. 4\left(\frac{1}{2} pr\right) \left(c + (t + b)(\sqrt{2} - 2) \right) (\sqrt{2}) + 16\left(\frac{1}{3} r\left(\frac{1}{2} pr(\sqrt{2} - 1)\right)\right) \right] - \\
& \left[2\left(\frac{1}{2} \frac{r}{p} (p - d)^2\right) \left(L - 2\left(c + \left(t + b - \frac{rd}{p} \right) (\sqrt{2} - 1) \right) \right) + \right. \\
& \left. 2\left(\frac{1}{2} \frac{r}{p} (p - d)^2\right) \left(W - 2\left(c + \left(t + b - \frac{rd}{p} \right) (\sqrt{2} - 1) \right) \right) + \right. \\
& \left. 4\left(\frac{1}{2} \frac{r}{p} (p - d)^2\right) \left(c + \left(t + b - \frac{rd}{p} \right) (\sqrt{2} - 2) \right) (\sqrt{2}) + \right. \\
& \left. 16\left(\frac{1}{3} \frac{r}{p} (p - d)\right) \left(\frac{1}{2} \frac{r}{p} (p - d)^2 (\sqrt{2} - 1)\right) \right].
\end{aligned}$$

After some simplification, the equation for approximating the penetrated volume of the CRB base section can be written as

$$\begin{aligned}
V_{pen,6} = & (d) \left[(L - 2(t + b))(W - 2(t + b)) - 2(c + (t + b)(\sqrt{2} - 2))^2 \right] + \tag{A.6} \\
& pr \left[L + W - 4(c + (t + b)(\sqrt{2} - 1)) + \right. \\
& \left. 2\sqrt{2}(c + (t + b)(\sqrt{2} - 2)) + \frac{8}{3} r(\sqrt{2} - 1) \right] - \\
& \frac{r}{p} (p - d)^2 \left[L + W - 4\left(c + \left(t + b - \frac{rd}{p} \right) (\sqrt{2} - 1) \right) + \right. \\
& \left. 2\sqrt{2}\left(c + \left(t + b - \frac{rd}{p} \right) (\sqrt{2} - 2) \right) + \frac{8}{3} \frac{r}{p} (p - d) (\sqrt{2} - 1) \right] \\
= & (d) \left[(L - 2(t + b))(W - 2(t + b)) - 2(c + (t + b)(\sqrt{2} - 2))^2 \right] + \\
& \frac{d^2}{h_s} \left[(L + W + (1 - \sqrt{2})(2c\sqrt{2} + 8(t + b))) \left(2\sqrt{h_s^2 + b^2} - b \right) + \right.
\end{aligned}$$

$$\frac{8db}{h_5} \left(1 - \sqrt{2} \left(\frac{2b}{3} - \sqrt{h_5^2 + b^2} \right) \right) \Bigg]$$

where

- $V_{full,6}$ = the approximate full volume of the CRB base section
 - $V_{pen,6}$ = the approximate penetrated volume of the CRB base section
 - r = the horizontal penetration distance below the angled portion of the bottom section of the refractory wall and into the CRB base
 - p = the horizontal penetration distance below the angled portion of the bottom section of the refractory wall and into the CRB base
- and other quantities are as defined previously.

In Figure A.4, r and p are labeled relative to the approximation of the penetrated volume of the CRB base. Note that r and p used to approximate the penetrated volume of the base are equal to the quantities r and p used to approximate the penetrated volume of the bottom wall section; therefore, the same labels are used in both cases. The volume $V_{full,6}$ in Eq. (A.5) is an approximation because it is based on the assumption that the CRB base section has uniform thickness t_b and overall dimensions represented by L , W , and c . The volume $V_{pen,6}$ in Eq. (A.6) is an approximation because it is based on 1) an estimate of the penetration depth assuming that the penetration depth is uniform over the refractory base and the region penetrated below the angled portion of the bottom wall section is accurately represented by r and p , and 2) on the nominal value for the width of the angled portion of the bottom section of the refractory wall. Furthermore, the actual CRB base includes several sump regions that are not depicted in the figures presented in this report and are not represented in Eq. (A.5) and (A.6).

To provide some intuitive assessment of the equations used to calculate penetrated volume, the equations were based on different nominal penetration depths. Figure A.5 shows the approximate total penetrated volumes obtained by summing calculated penetrated volumes from Eq. (A.2), (A.4), and (A.6) for penetration depths ranging from 0.25 to 6 inches and plotting the against these penetration depths.

Figures A.6 through A.8 provide further clarification of the general CRB configuration. All figures are simplifications of AMEC ICV box refractory assembly drawings such as F-145579-35-D-0016 and F-145579-35-D-0004. The AMEC drawings show additional details such as the sump areas in the CRB base and the slight taper in the CRB walls.

A.2.5 Volume Approximation Uncertainties

Several options could be used to generate estimates of the uncertainty associated with the volume approximations (both full and penetrated) for the different sections of the CRB. One option is error propagation. However, the volume approximation formulas are somewhat complicated in that they involve numerous variables that should be considered as having uncertainty (L , W , h_i , t , d , b , and c). Therefore, error propagation (even first-order error propagation) would result in very complicated formulas for estimating the uncertainties associated with the volume calculations. Furthermore, applying error propagation to the volume approximation formulas would require repeated use of error propagation

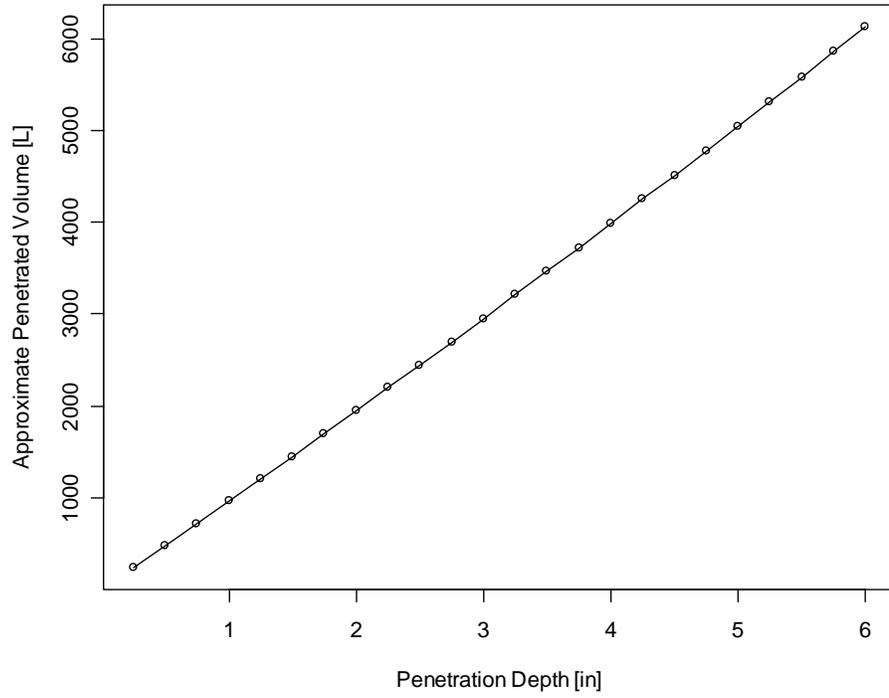


Figure A.5. Plot of Penetration Depths and Approximate Penetrated Volumes

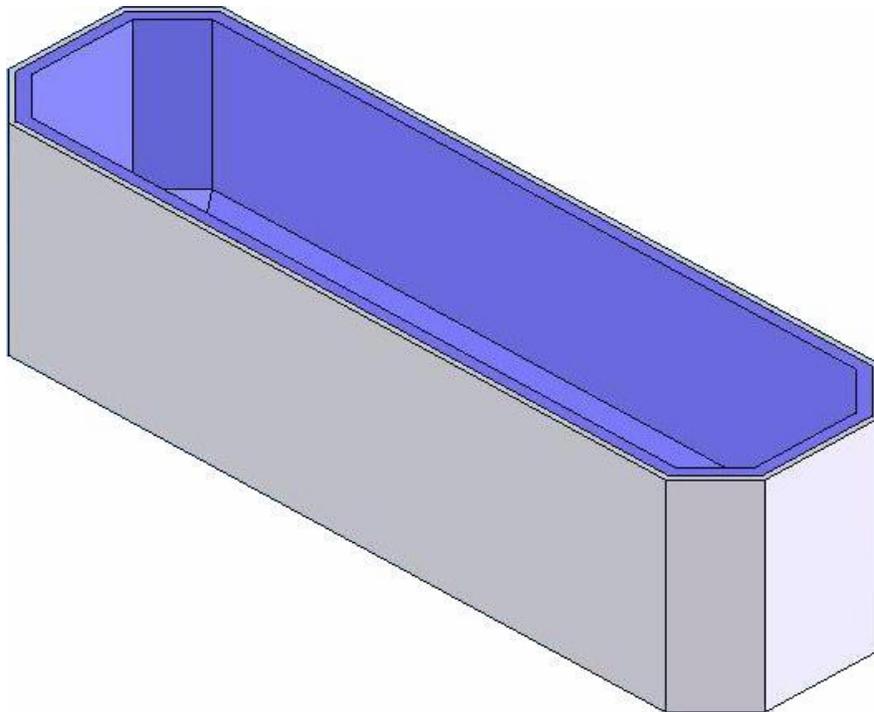


Figure A.6. Full View of CRB

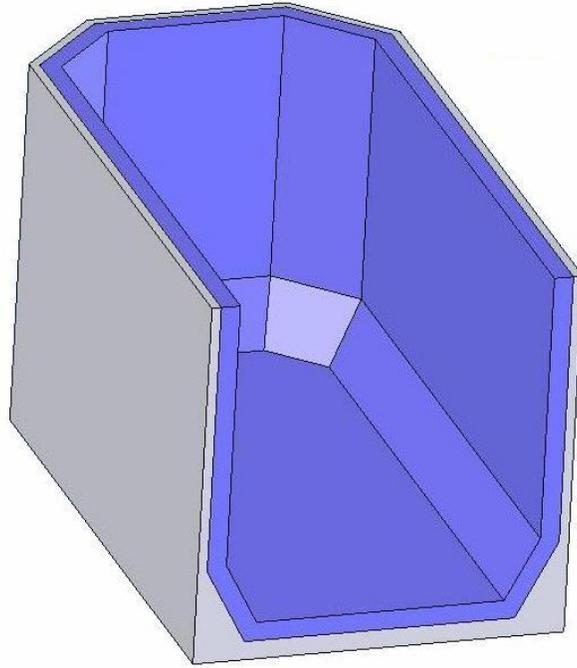


Figure A.7. Cutaway View of CRB

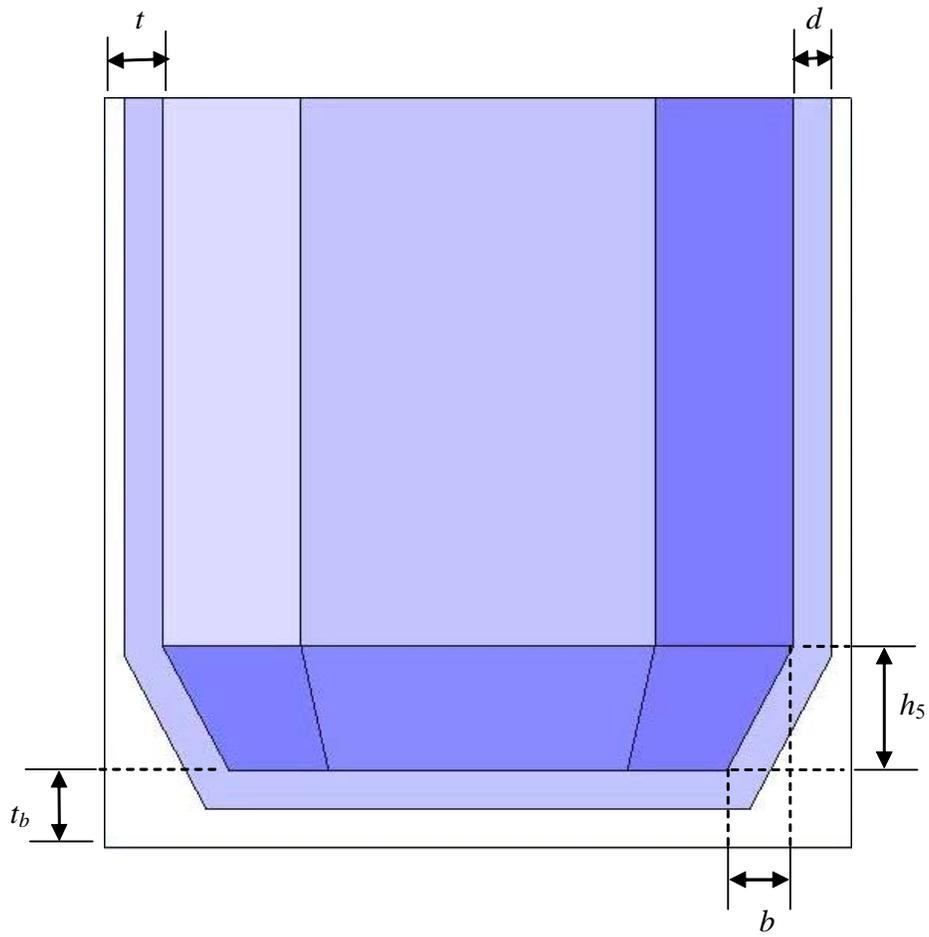


Figure A.8. End View of CRB

formulas to estimate the uncertainties associated with the variables r and p , and those uncertainties would be used to estimate the uncertainties associated with the volumes. For these reasons, using simple error propagation formulas (first-order not involving covariance terms) to estimate the uncertainties associated with volume approximations could be inappropriate. Furthermore, the use of more accurate error propagation formulas (higher order with covariance terms) in connection with the volume approximation formulas in Eq. (A.1) through (A.6) would be impractical. However, to obtain some indication of the magnitude of volume uncertainty that might result from error propagation methods, error propagation was used in simpler volume approximation formulas that assumed a rectangular refractory. The volume determined by these formulas was within 5% of that determined using Eq. (A.1) through (A.6), and the error propagation methods suggested that uncertainty associated with the volume approximations could be represented by a relative standard deviation of about 10%. The effect of a 10% relative uncertainty in the volume approximation on the uncertainty associated with the Re fraction and Re scale-up factor is addressed as part of the third option discussed below.

A second option that could be used to estimate uncertainties associated with the volume approximations is the use of a Monte Carlo simulation. For this option, various nominal values would be assigned for each of the input variables as well as various nominal values for their corresponding uncertainties. A computer simulation would then be conducted wherein the uncertainty associated with the volume approximations would be calculated for each possible combination of input variable values and their corresponding uncertainties. This option was investigated using several nominal values (considered to be reasonable) for the uncertainties associated with the different input variables. The simulation indicated that the most important quantity affecting the estimates of volume uncertainty was the uncertainty associated with the penetration depth. Thus, it is important to have an accurate approximation of penetration depth uncertainty in order to accurately quantify volume uncertainty. Other input variables to the volume approximation equation could be determined (or specified for the simulation approach) with less accuracy because they had little impact on volume uncertainty. Furthermore, the relative uncertainty in the overall penetrated volume approximation was found to be (via simulation) only slightly higher than the nominal uncertainty assigned to the penetration depth. Thus, using a nominal penetration depth uncertainty of 10% resulted in a relative uncertainty in the overall penetrated volume approximation of just over 10%, and a nominal penetration depth uncertainty of 20% resulted in a relative uncertainty in the overall penetrated volume approximation of about 21%. A relative uncertainty of 10 to 20% seems reasonable (achievable) for representing penetration depth uncertainty. The effect of a 10 to 20% relative uncertainty in the volume approximation on the uncertainty associated with the Re fraction and Re scale-up factor is addressed as part of the third option discussed below.

A third option for estimating the uncertainties associated with the volume approximations is to assign several nominal values, use them in the equations that estimate the uncertainty associated with the Re fraction and scale-up factor, and see how these final uncertainties are affected by the nominal values for volume uncertainty. This option was investigated using nominal relative standard deviation percentages of 5, 10, 20, and 30% for the uncertainties associated with the volume approximations. That is, the estimate of uncertainty associated with the approximate volume by section (denoted as σ_{V_i} in Eq. A.10) was calculated using $\sigma_{V_i} = AV_i$, where A was 5, 10, 20, or 30%, and V_i represents the approximate volume for section i based on either full-volume or penetrated-volume calculations. As mentioned previously, a relative volume uncertainty of 10–20% seems reasonable based on rough error propagation estimates and simulation. Lower (5%) and higher (30%) nominal values were also considered for

completeness. This range of nominal relative volume uncertainty values resulted in very minimal differences in the uncertainty associated with the final Re fraction and scale-up factor estimates. More specifically, when nominal volume uncertainties of 5, 10, 20, or 30% times the volume approximations from Eq. (A.1) through (A.6) were used as estimates of volume uncertainty, the corresponding estimated relative uncertainties in the Re fraction estimate ranged from 23.28 to 28.72% for full volume (assuming no adjustment in Re concentrations due to background extractable Re in blank samples) and from 20.27 to 27.01% for the 6-inch penetrated volume. Similarly, estimated relative uncertainties in the Re scale-up factor estimate ranged from 42.26 to 45.48% for full-volume cases and from 40.68 to 44.42% for 6-inch penetrated volume cases (see Table 5 of the main report for details). Thus, increases in volume uncertainty do not cause similar increases in Re fraction uncertainty. Based on the options discussed above, particularly the second and third options, using a nominal relative uncertainty of 10% of the volume approximations for the refractory sections would probably be reasonable.

A.2.6 Specific Extractable Re Mass

Specific extractable Re mass (mass of extractable Re divided by mass of refractory fragment with units of $\mu\text{g}/\text{kg}$) is an estimated input variable. It is estimated by first calculating a weighted average of analyzed specific extractable Re masses over the fragments of a given sample from the CRB (the weighting is based on fragment masses), then averaging over the samples within a given vertical section of the CRB. The underlying assumption is that the specific extractable Re mass is homogeneous within a given vertical section of the CRB but not necessarily over different vertical sections. Thus, specific extractable Re mass estimates are calculated for each section of the CRB. The uncertainty estimates associated with specific extractable Re masses are also calculated for each section of the CRB. These uncertainty estimates are standard deviations calculated using the weighted averages of specific extractable Re mass for the samples taken from a given section of the CRB.

Estimated specific extractable masses (c_{ij} with units of $\mu\text{g}/\text{kg}$) and their respective estimated uncertainties ($\sigma_{\hat{c}_{ij}}$) are supplied by the laboratory following analysis of samples from FS-38B. The subscript i represents the vertical section of the CRB, and the subscript j represents the j^{th} sample from section i . For each section of the CRB, average specific extractable Re masses and the uncertainty associated with the average specific extractable Re masses are calculated using the following equations:

$$\bar{c}_i = \frac{\sum_{j=1}^{N_{\text{samples},i}} c_{ij}}{N_{\text{samples},i}} \quad \text{and} \quad (A.7)$$

$$\sigma_{\bar{c}_i} = \sqrt{\frac{\sum_{j=1}^{N_{\text{samples},i}} (c_{ij} - \bar{c}_i)^2}{N_{\text{samples},i} (N_{\text{samples},i} - 1)}} \quad (A.8)$$

where \bar{c}_i (with units of $\mu\text{g}/\text{kg}$) denotes the estimated average specific extractable Re mass for section i , $\sigma_{\bar{c}_i}$ denotes the estimated standard deviation,^(a) or estimated standard error, in the average specific extractable Re mass for section i , and $N_{\text{samples},i}$ denotes the number of samples taken from section i . The quantity \bar{c}_i from Eq. (A.7) is an estimate of the specific extractable Re mass for section i because it is calculated using specific extractable Re mass estimates obtained from several sampling locations within section i . The same is true of the quantity $\sigma_{\bar{c}_i}$ from Eq. (A.8).

It is important to point out that standard deviations calculated using Eq. (A.8) account for sources of uncertainty represented in the sampling and analysis process used to generate the estimated specific extractable masses (c_{ij}). Thus, these standard deviations include fragment-to-fragment uncertainty, sampling location uncertainty, and short-term analytical uncertainty. However, because the analyses were conducted over a relatively short period of time and at a single lab, long-term and lab-to-lab analytical uncertainties are not included in the standard deviations obtained using Eq. (A.8). The standard deviations would probably be considerably larger if those uncertainties were present in the analytical process. That is, sampling and analysis methods must be taken into consideration when interpreting or using the standard deviations obtained using Eq. (A.8). Calculated standard deviation values should not be considered universal for representing all sampling and analysis methods. This could be an important consideration if analysis methods change in the future.

A.2.7 Refractory Density

A specified nominal value is assigned for the refractory density input variable. The density, ρ_{refract} , is assumed to be the same over all CRB sections. The uncertainty associated with refractory density, $\sigma_{\rho_{\text{refract}}}$, is a specified relative amount that is considered constant for all sections of the CRB. The nominal value for refractory density is 2611 g/L (calculations were actually conducted using 26.11.00953 g/L, or 163 lb/ft³). This value is taken from vendor information.^(b) The uncertainty associated with the refractory density is assumed to be $\sigma_{\rho_{\text{refract}}} = 0.05\rho_{\text{refract}}$.

A.2.8 Re Inventory

A specified nominal value is assigned for the Re inventory input variable. This nominal value represents the total amount of Re spiked into the waste simulant used in FS-38B and applies to the overall test. The Re inventory is denoted m_{total} . The uncertainty associated with the Re inventory, $\sigma_{m_{\text{total}}}$, is a specified relative amount that also applies to the overall test. For FS-38B, m_{total} is estimated to be 229.52 g, and it is assumed that $\sigma_{m_{\text{total}}} = 0.10m_{\text{total}}$.

(a) In most statistical formulas, the notation σ is used to denote a true (known without uncertainty) standard deviation value, and the notation $\hat{\sigma}$ is used to denote an estimate of this standard deviation. However, for this report the “hat” is omitted from the sigma in Equation (A.8), and in subsequent standard deviation equations. This convention is adopted to simplify notation because many of the variables that appear in the equations herein are approximations or estimates, and would therefore include hats.

(b) Spec sheet for Vibrocast 60PC from Resco Products, Inc, 2 Penn Center West, Suite 430, Pittsburgh, PA, <http://www.rescoproducts.com/>.

A.3 Calculated Quantities of Interest

A.3.1 Extractable Re Mass

Volume approximations (either full or penetrated), specific extractable Re mass estimates, and the nominal (or estimated) refractory density are used to calculate estimates of the extractable Re mass (also called the soluble Re mass) contained in each section of the CRB using the equation:

$$m_i = V_i \bar{c}_i \rho_{refract} \quad (\text{A.9})$$

The uncertainty associated with these estimates of extractable Re mass (by section) is calculated using the equation:

$$\sigma_{m_i} = m_i \left[\frac{\sigma_{V_i}^2}{V_i^2} + \frac{\sigma_{\bar{c}_i}^2}{\bar{c}_i^2} + \frac{\sigma_{\rho_{refract}}^2}{\rho_{refract}^2} \right]^{\frac{1}{2}} \quad (\text{A.10})$$

In Eq. (A.9) and (A.10), m_i denotes the estimated extractable Re mass for section i , σ_{m_i} is the estimated standard deviation in the extractable Re mass for section i , V_i is the approximate volume for section i (based on full- or penetrated-volume approximation equations), and σ_{V_i} is the estimated standard deviation describing the uncertainty associated with the approximate volume for section i . Other quantities are as defined previously. The quantity m_i from Eq. (A.9) is an estimate of the extractable Re mass for section i because it is calculated using an estimate of specific extractable Re mass, a volume approximation, and a nominal density value. The same is true for σ_{m_i} in Eq. (A.10), which also includes estimates of the uncertainties associated with the estimates of specific extractable Re mass, volume approximations, and the nominal density. Furthermore, Eq. (A.10) is actually an approximation formula obtained by applying first-order error propagation methods given in Eq. (A.22) and subsequently to Eq. (A.9).

The estimates of extractable Re mass by section are summed to obtain an estimate for the extractable Re mass contained in the entire CRB, as shown in the equation

$$m = \sum_{i=1}^{N_{sections}} m_i \quad (\text{A.11})$$

The uncertainty in the overall estimate from Eq. (A.11) is calculated using the equation:

$$\sigma_m = \left[\frac{m^2 \sigma_{\rho_{refract}}^2}{\rho_{refract}^2} + \sum_{i=1}^{N_{sections}} m_i^2 \left(\frac{\sigma_{V_i}^2}{V_i^2} + \frac{\sigma_{\bar{c}_i}^2}{\bar{c}_i^2} \right) \right]^{\frac{1}{2}} \quad (\text{A.12})$$

In Eq. (A.11) and (A.12), m denotes the estimated total extractable Re mass over the entire CRB, σ_m denotes the estimated standard deviation in the total extractable Re mass, $N_{sections}$ denotes the number of sections represented in the CRB, and the other quantities are as defined previously. The quantities m and σ_m are estimates of the total extractable Re mass for the CRB and its corresponding uncertainty because they are calculated using estimates of the extractable Re mass for each section and corresponding uncertainty estimates. Eq. (A.12) is based on the simplifying assumption that the estimates of extractable Re mass by section (m_i) are statistically independent. A more complicated equation involving covariances could be used in place of Eq. (A.12) if future results indicate that the independence assumption is not justified and covariance estimates can be determined.

A.3.2 Re Fraction

The fraction of the Re inventory that is ultimately absorbed into the CRB is calculated by dividing the overall mass of extractable Re in the CRB by the total Re inventory as follows

$$f = \frac{m}{m_{total}} \quad (\text{A.13})$$

The uncertainty associated with the Re fraction estimate is calculated using the equation:

$$\sigma_f = f \left[\frac{\sigma_m^2}{m^2} + \frac{\sigma_{m_{total}}^2}{m_{total}^2} \right]^{\frac{1}{2}} \quad (\text{A.14})$$

In Eq. (A.13) and (A.14), f denotes the estimated Re fraction for the CRB for FS-38B, σ_f denotes the estimated standard deviation in the Re fraction for FS-38B, m_{total} denotes the total Re inventory spiked into FS-38B, $\sigma_{m_{total}}$ denotes the estimated standard deviation in the total Re inventory, and other quantities are as defined previously. As discussed, it is assumed that $\sigma_{m_{total}} = 0.10m_{total}$. The quantities f and σ_f are estimates of the Re fraction for the CRB and its corresponding uncertainty because they are calculated using estimates of the total Re mass absorbed into the CRB, the total Re inventory for FS-38B, and corresponding uncertainty estimates. Furthermore, Eq. (A.14) is actually an approximation formula obtained by applying the first-order error propagation methods in Eq. (A.21) to Eq. (A.13).

Table A.1 provides quantities used to estimate the Re fraction for test FS-38B. Information is given for each vertical section of the CRB based on the specified background extractable Re adjustment amounts. The Re concentrations listed for each section are obtained by averaging the adjusted Re concentrations over the samples of a given section. As explained in Section 5 of the main report, the adjusted Re concentrations are obtained by subtracting the specified adjustment amounts from the analyzed Re concentrations for each CRB sample collected. The Re concentrations listed under the zero adjustment case in Table A.1 are the means by section of the sample averages of analyzed Re concentrations given in Table 3 (Section 4.3). Because the Re concentrations given in Table A.1 are mean values, their

Table A.1. Re Fraction Estimates from Test FS-38B

Background Adjustment Amt (µg/kg)	Vertical Section	Section Volume (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass Total (g)	Re Fraction
0	1	1675.41	14.89	7.91	0.065	1.71	0.0075
	2	725.29	31.05	8.45	0.059		
	3	574.77	573.00	0.00	0.860		
	4	898.67	145.85	87.15	0.342		
	5	1280.90	27.70	1.15	0.093		
	6	2737.30	41.26	38.94	0.295		
1	1	1675.41	13.89	7.91	0.061	1.69	0.0074
	2	725.29	30.05	8.45	0.057		
	3	574.77	572.00	0.00	0.858		
	4	898.67	144.85	87.15	0.340		
	5	1280.90	26.70	1.15	0.089		
	6	2737.30	40.26	38.94	0.288		
10	1	1675.41	6.40	6.40	0.028	1.54	0.0067
	2	725.29	21.05	8.45	0.040		
	3	574.77	563.00	0.00	0.845		
	4	898.67	135.85	87.15	0.319		
	5	1280.90	17.70	1.15	0.059		
	6	2737.30	35.10	35.10	0.251		
20	1	1675.41	1.40	1.40	0.006	1.39	0.0061
	2	725.29	11.05	8.45	0.021		
	3	574.77	553.00	0.00	0.830		
	4	898.67	125.85	87.15	0.295		
	5	1280.90	7.70	1.15	0.026		
	6	2737.30	30.10	30.10	0.215		
30	1	1675.41	0.00	0.00	0.000	1.28	0.0056
	2	725.29	4.75	4.75	0.009		
	3	574.77	543.00	0.00	0.815		
	4	898.67	115.85	87.15	0.272		
	5	1280.90	0.00	0.00	0.000		
	6	2737.30	25.10	25.10	0.179		
40	1	1675.41	0.00	0.00	0.000	1.19	0.0052
	2	725.29	0.00	0.00	0.000		
	3	574.77	533.00	0.00	0.800		
	4	898.67	105.85	87.15	0.248		
	5	1280.90	0.00	0.00	0.000		
	6	2737.30	20.10	20.10	0.144		

corresponding standard deviations (also listed in Table A.1) are actually standard errors. The Re masses listed in Table A.1 are obtained by multiplying the adjusted Re concentrations by the corresponding volume approximations (Table A.1), then multiplying the resulting product by the nominal refractory density value of 2611 g/L. Summing these Re masses over the six CRB sections yields the total Re mass estimates given in Table A.1 for each of the adjustment amount scenarios. Dividing the total Re mass estimates by the Re loading for test FS-38B (229.52 g) yields the Re fraction estimates given in Table A.1 for each of the adjustment amount scenarios. This series of calculations is specified by Eq. (A.9), (A.11), and (A.13). The estimates of Re fraction are used to estimate the Re scale-up factor for test FS-38B (see Table 8 in Section 5.2). The estimates of total Re mass and Re fraction given in Table A.1 also appear in Table 7 (Section 5.2) of the main report.

A.3.3 Re Scale-Up Factor

The Re scale-up factor is the ratio calculated by dividing the Re fraction for ES tests by the Re fraction for FS tests. That is,

$$\zeta = \frac{f_{\text{Re},ES}}{f} \quad (\text{A.15})$$

The uncertainty associated with the Re scale-up factor is calculated using the equation:

$$\sigma_{\zeta} = \zeta \left[\frac{\sigma_{f_{\text{Re},ES}}^2}{f_{\text{Re},ES}^2} + \frac{\sigma_f^2}{f^2} \right]^{\frac{1}{2}} \quad (\text{A.16})$$

In Eq. (A.15) and (A.16), ζ denotes the estimated Re scale-up factor, σ_{ζ} denotes the estimated standard deviation for the Re scale-up factor, $f_{\text{Re},ES}$ denotes the estimated Re fraction for the CRB of ES tests, $\sigma_{f_{\text{Re},ES}}$ denotes the estimated standard deviation in the Re fraction for ES tests, and other quantities are as defined previously. The quantities ζ and σ_{ζ} are estimates of the Re scale-up factor and its corresponding uncertainty because they are calculated using estimates of the Re fraction for the CRB of FS-38B, the Re fraction for the CRB of ES tests, and corresponding uncertainty estimates. Furthermore, Eq. (A.16) is an approximation formula obtained by applying first-order error propagation methods (given in Eq. A.21) to Eq. (A.15).

The quantities $f_{\text{Re},ES}$ and $\sigma_{f_{\text{Re},ES}}$ in Equations (A.15) and (A.16) are based on calculations conducted using existing ES test data. Table A.2 contains information similar to that presented in Table A.1 but relative to six specific ES tests; ES-31A, ES-31B, ES-31C, ES-31E, ES-32A, and ES-32B. Data were also available from ES-31D, but the waste simulant used for that test was not considered representative of that used in other ES and current FS tests. Therefore, the data from ES-31D was not included in the calculations presented in this report. The different pages of Table A.2 show results for the six ES tests based on background adjustments of 0, 1, 10, 20, 30, and 40 $\mu\text{g}/\text{kg}$, the same adjustment amounts mentioned previously relative the calculations for FS-38B. In each of the adjustment cases, the quantity $f_{\text{Re},ES}$ is the average of the Re fraction estimated from the six ES tests listed in Table A.2, and $\sigma_{f_{\text{Re},ES}}$ is

Table A.2. Re Fraction Estimates from ES Tests

Background Adjustment Amount = 0 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	773.37	552.42	0.065	0.117	7.715	0.015
	2	20.19	578.28	508.37	0.034			
	3	20.19	166.51	29.49	0.010			
	4	17.30	165.12	33.45	0.008			
	5	17.17	6.98	5.03	0.000			
ES-31B	1	23.07	13.27	2.19	0.001	0.003	0.413	0.008
	2	23.07	9.10	0.93	0.001			
	3	28.84	14.79	2.41	0.001			
	4	11.54	10.61	3.80	0.000			
	5	17.17	1.90	0.83	0.000			
ES-31C	1	23.07	135.37	77.97	0.009	0.046	0.796	0.057
	2	17.30	66.11	23.67	0.003			
	3	17.30	312.28	61.13	0.016			
	4	17.30	205.09	16.55	0.010			
	5	17.17	147.95	45.03	0.007			
ES-31E	1	17.30	96.47	24.46	0.005	0.015	0.763	0.020
	2	23.07	40.29	0.00	0.003			
	3	23.07	76.30	0.00	0.005			
	4	23.07	33.68	0.00	0.002			
	5	17.17	1.41	0.00	0.000			
ES-32A	1	23.07	465.06	0.00	0.031	0.116	7.510	0.015
	2	23.07	337.89	0.00	0.023			
	3	23.07	358.51	0.00	0.024			
	4	17.30	103.62	0.00	0.005			
	5	17.17	673.63	0.00	0.034			
ES-32B	1	17.30	84.43	12.26	0.004	0.008	0.709	0.011
	2	23.07	33.20	18.60	0.002			
	3	23.07	11.02	2.27	0.001			
	4	23.07	7.94	4.79	0.001			
	5	17.17	5.89	0.00	0.000			
Mean								0.021
Std. Error								0.0074

Table A.2 (contd)

Background Adjustment Amount = 1 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	772.37	552.42	0.065	0.117	7.715	0.015
	2	20.19	577.28	508.37	0.034			
	3	20.19	165.51	29.49	0.010			
	4	17.30	164.12	33.45	0.008			
	5	17.17	5.98	5.03	0.000			
ES-31B	1	23.07	12.27	2.19	0.001	0.003	0.413	0.007
	2	23.07	8.10	0.93	0.001			
	3	28.84	13.79	2.41	0.001			
	4	11.54	9.61	3.80	0.000			
	5	17.17	1.00	0.83	0.000			
ES-31C	1	23.07	134.37	77.97	0.009	0.045	0.796	0.057
	2	17.30	65.11	23.67	0.003			
	3	17.30	311.28	61.13	0.016			
	4	17.30	204.09	16.55	0.010			
	5	17.17	146.95	45.03	0.007			
ES-31E	1	17.30	95.47	24.46	0.005	0.015	0.763	0.019
	2	23.07	39.29	0.00	0.003			
	3	23.07	75.30	0.00	0.005			
	4	23.07	32.68	0.00	0.002			
	5	17.17	0.41	0.00	0.000			
ES-32A	1	23.07	464.06	0.00	0.031	0.116	7.510	0.015
	2	23.07	336.89	0.00	0.023			
	3	23.07	357.51	0.00	0.024			
	4	17.30	102.62	0.00	0.005			
	5	17.17	672.63	0.00	0.033			
ES-32B	1	17.30	83.43	12.26	0.004	0.008	0.709	0.011
	2	23.07	32.20	18.60	0.002			
	3	23.07	10.02	2.27	0.001			
	4	23.07	6.94	4.79	0.000			
	5	17.17	4.89	0.00	0.000			
Mean								0.021
Std. Error								0.0075

Table A.2 (contd)

Background Adjustment Amount = 10 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	763.37	552.42	0.064	0.114	7.715	0.015
	2	20.19	568.28	508.37	0.033			
	3	20.19	156.51	29.49	0.009			
	4	17.30	155.12	33.45	0.008			
	5	17.17	1.00	5.03	0.000			
ES-31B	1	23.07	3.91	2.19	0.000	0.001	0.413	0.002
	2	23.07	0.46	0.93	0.000			
	3	28.84	4.87	2.41	0.000			
	4	11.54	3.05	3.80	0.000			
	5	17.17	0.00	0.83	0.000			
ES-31C	1	23.07	125.37	77.97	0.008	0.043	0.796	0.054
	2	17.30	56.11	23.67	0.003			
	3	17.30	302.28	61.13	0.015			
	4	17.30	195.09	16.55	0.010			
	5	17.17	137.95	45.03	0.007			
ES-31E	1	17.30	86.47	24.46	0.004	0.012	0.763	0.016
	2	23.07	30.29	0.00	0.002			
	3	23.07	66.30	0.00	0.004			
	4	23.07	23.68	0.00	0.002			
	5	17.17	0.00	0.00	0.000			
ES-32A	1	23.07	455.06	0.00	0.030	0.113	7.510	0.015
	2	23.07	327.89	0.00	0.022			
	3	23.07	348.51	0.00	0.023			
	4	17.30	93.62	0.00	0.005			
	5	17.17	663.63	0.00	0.033			
ES-32B	1	17.30	74.43	12.26	0.004	0.005	0.709	0.008
	2	23.07	23.20	18.60	0.002			
	3	23.07	1.65	2.27	0.000			
	4	23.07	1.37	4.79	0.000			
	5	17.17	0.00	0.00	0.000			
Mean								0.018
Std. Error								0.0075

Table A.2 (contd)

Background Adjustment Amount = 20 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	753.37	552.42	0.063	0.111	7.715	0.014
	2	20.19	558.28	508.37	0.033			
	3	20.19	146.51	29.49	0.009			
	4	17.30	145.12	33.45	0.007			
	5	17.17	0.00	5.03	0.000			
ES-31B	1	23.07	0.22	2.19	0.000	0.000	0.413	0.000
	2	23.07	0.00	0.93	0.000			
	3	28.84	0.00	2.41	0.000			
	4	11.54	1.05	3.80	0.000			
	5	17.17	0.00	0.83	0.000			
ES-31C	1	23.07	115.37	77.97	0.008	0.040	0.796	0.051
	2	17.30	46.11	23.67	0.002			
	3	17.30	292.28	61.13	0.015			
	4	17.30	185.09	16.55	0.009			
	5	17.17	127.95	45.03	0.006			
ES-31E	1	17.30	76.47	24.46	0.004	0.010	0.763	0.013
	2	23.07	20.29	0.00	0.001			
	3	23.07	56.30	0.00	0.004			
	4	23.07	13.68	0.00	0.001			
	5	17.17	0.00	0.00	0.000			
ES-32A	1	23.07	445.06	0.00	0.030	0.110	7.510	0.015
	2	23.07	317.89	0.00	0.021			
	3	23.07	338.51	0.00	0.023			
	4	17.30	83.62	0.00	0.004			
	5	17.17	653.63	0.00	0.033			
ES-32B	1	17.30	64.43	12.26	0.003	0.004	0.709	0.006
	2	23.07	15.90	18.60	0.001			
	3	23.07	0.00	2.27	0.000			
	4	23.07	0.00	4.79	0.000			
	5	17.17	0.00	0.00	0.000			
Mean								0.016
Std. Error								0.0072

Table A.2 (contd)

Background Adjustment Amount = 30 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	743.37	552.42	0.062	0.109	7.715	0.014
	2	20.19	548.28	508.37	0.032			
	3	20.19	136.51	29.49	0.008			
	4	17.30	135.12	33.45	0.007			
	5	17.17	0.00	5.03	0.000			
ES-31B	1	23.07	0.00	2.19	0.000	0.000	0.413	0.000
	2	23.07	0.00	0.93	0.000			
	3	28.84	0.00	2.41	0.000			
	4	11.54	0.00	3.80	0.000			
	5	17.17	0.00	0.83	0.000			
ES-31C	1	23.07	105.37	77.97	0.007	0.038	0.796	0.047
	2	17.30	36.11	23.67	0.002			
	3	17.30	282.28	61.13	0.014			
	4	17.30	175.09	16.55	0.009			
	5	17.17	118.10	45.03	0.006			
ES-31E	1	17.30	66.47	24.46	0.003	0.007	0.763	0.010
	2	23.07	10.29	0.00	0.001			
	3	23.07	46.30	0.00	0.003			
	4	23.07	3.68	0.00	0.000			
	5	17.17	0.00	0.00	0.000			
ES-32A	1	23.07	435.06	0.00	0.029	0.107	7.510	0.014
	2	23.07	307.89	0.00	0.021			
	3	23.07	328.51	0.00	0.022			
	4	17.30	73.62	0.00	0.004			
	5	17.17	643.63	0.00	0.032			
ES-32B	1	17.30	54.43	12.26	0.003	0.003	0.709	0.005
	2	23.07	10.90	18.60	0.001			
	3	23.07	0.00	2.27	0.000			
	4	23.07	0.00	4.79	0.000			
	5	17.17	0.00	0.00	0.000			
Mean								0.015
Std. Error								0.0068

Table A.2 (contd)

Background Adjustment Amount = 40 (µg/kg)								
Test	Vertical Section	Section Vol. (L)	Re Conc. (µg/kg)	SD Re Conc. (µg/kg)	Re Mass by Section (g)	Re Mass by Test (g)	Re Loading	Re Fraction
ES-31A	1	28.84	733.37	552.42	0.061	0.106	7.715	0.014
	2	20.19	538.28	508.37	0.031			
	3	20.19	126.51	29.49	0.007			
	4	17.30	125.12	33.45	0.006			
	5	17.17	0.00	5.03	0.000			
ES-31B	1	23.07	0.00	2.19	0.000	0.000	0.413	0.000
	2	23.07	0.00	0.93	0.000			
	3	28.84	0.00	2.41	0.000			
	4	11.54	0.00	3.80	0.000			
	5	17.17	0.00	0.83	0.000			
ES-31C	1	23.07	95.37	77.97	0.006	0.035	0.796	0.044
	2	17.30	26.11	23.67	0.001			
	3	17.30	272.28	61.13	0.014			
	4	17.30	165.09	16.55	0.008			
	5	17.17	110.60	45.03	0.006			
ES-31E	1	17.30	56.47	24.46	0.003	0.005	0.763	0.007
	2	23.07	0.29	0.00	0.000			
	3	23.07	36.30	0.00	0.002			
	4	23.07	0.00	0.00	0.000			
	5	17.17	0.00	0.00	0.000			
ES-32A	1	23.07	425.06	0.00	0.028	0.104	7.510	0.014
	2	23.07	297.89	0.00	0.020			
	3	23.07	318.51	0.00	0.021			
	4	17.30	63.62	0.00	0.003			
	5	17.17	633.63	0.00	0.032			
ES-32B	1	17.30	44.43	12.26	0.002	0.003	0.709	0.004
	2	23.07	5.90	18.60	0.000			
	3	23.07	0.00	2.27	0.000			
	4	23.07	0.00	4.79	0.000			
	5	17.17	0.00	0.00	0.000			
Mean								0.014
Std. Error								0.0065

the standard error of $f_{\text{Re},ES}$ which is the standard deviation of the six Re fraction estimates divided by the square root of 6 (the number of Re fraction values used in the calculation of $f_{\text{Re},ES}$). That is,

$$f_{\text{Re},ES} = \frac{\sum_{i=1}^6 f_{\text{Re},ES,i}}{6} \quad \text{and} \quad (\text{A.17})$$

$$\sigma_{f_{\text{Re},ES}} = \sqrt{\frac{\sum_{i=1}^6 (f_{\text{Re},ES,i} - f_{\text{Re},ES})^2}{6(6-1)}}. \quad (\text{A.18})$$

The mean Re fraction values given at the bottom of each page of Table A.2 are the $f_{\text{Re},ES}$ values specified in Eq. (A.15) to calculate the estimated Re scale-up factor values given in Tables 8 and 9 of the main report. The standard error values, also given at the bottom of each page of Table A.2, are the $\sigma_{f_{\text{Re},ES}}$ values used to calculate the estimated standard deviation values for the Re scale-up factor estimates given in Table 7 of the main report, as specified in Eq. (A.16). Because $\sigma_{f_{\text{Re},ES}}$ is a standard deviation calculated using various ES test Re fraction values, it includes test-to-test variation and uncertainty.

As explained relative to Table A.1, the Re concentrations listed in Table A.2 are obtained by subtracting the specified adjustment amounts from the analyzed Re concentrations for each sample collected, then averaging over the samples in each CRB section. The estimated Re masses by section listed in Table A.2 are obtained by multiplying the adjusted Re concentrations for each section by the corresponding CRB section volume, and then by the refractory density which is assumed to be 2897.86 g/L for each of the ES tests. Summing the Re mass estimates over the CRB sections yields the estimate of total Re mass for each ES test. Dividing the estimates of total Re mass by the specified Re loadings for the ES tests (given in Table A.2) yields the estimates of Re fraction for each ES test and each background adjustment case. This series of calculations is consistent with Eq. (A.9), (A.11), and (A.13).

A.3.4 Error Propagation

First-order error propagation methods are used to produce an estimate of the uncertainty associated with extractable Re mass estimates for each section and ultimately for the Re fraction and scale-up factor estimates for the CRB of FS-38B. According to error propagation methods, if Z is a function of the independent random variables X and Y , the variance of Z is approximated using

$$\sigma_Z^2 \approx \left(\frac{\partial Z}{\partial X}\right)^2 s_X^2 + \left(\frac{\partial Z}{\partial Y}\right)^2 s_Y^2 \quad (\text{A.19})$$

where s_X^2 denotes an estimate of the variance of X and s_Y^2 denotes an estimate of the variance of Y . Similarly, if Z is a function of the independent random variables W , X , and Y , then the variance of Z is approximated using

$$\sigma_Z^2 \approx \left(\frac{\partial Z}{\partial W}\right)^2 s_W^2 + \left(\frac{\partial Z}{\partial X}\right)^2 s_X^2 + \left(\frac{\partial Z}{\partial Y}\right)^2 s_Y^2 \quad (\text{A.20})$$

where s_W^2 denotes an estimate of the variance of W , s_X^2 denotes an estimate of the variance of X , and s_Y^2 denotes an estimate of the variance of Y . Thus, if $Z = X/Y$,

$$\sigma_Z^2 \approx \left(\frac{X}{Y}\right)^2 \left(\frac{s_X^2}{X^2} + \frac{s_Y^2}{Y^2}\right) \quad (\text{A.21})$$

If $Z = WXY$,

$$\sigma_Z^2 \approx (WXY)^2 \left(\frac{s_W^2}{W^2} + \frac{s_X^2}{X^2} + \frac{s_Y^2}{Y^2}\right) \quad (\text{A.22})$$

Eq. (A.21) and (A.22) are the two specific examples of error propagation formulas used in the uncertainty estimates presented previously.