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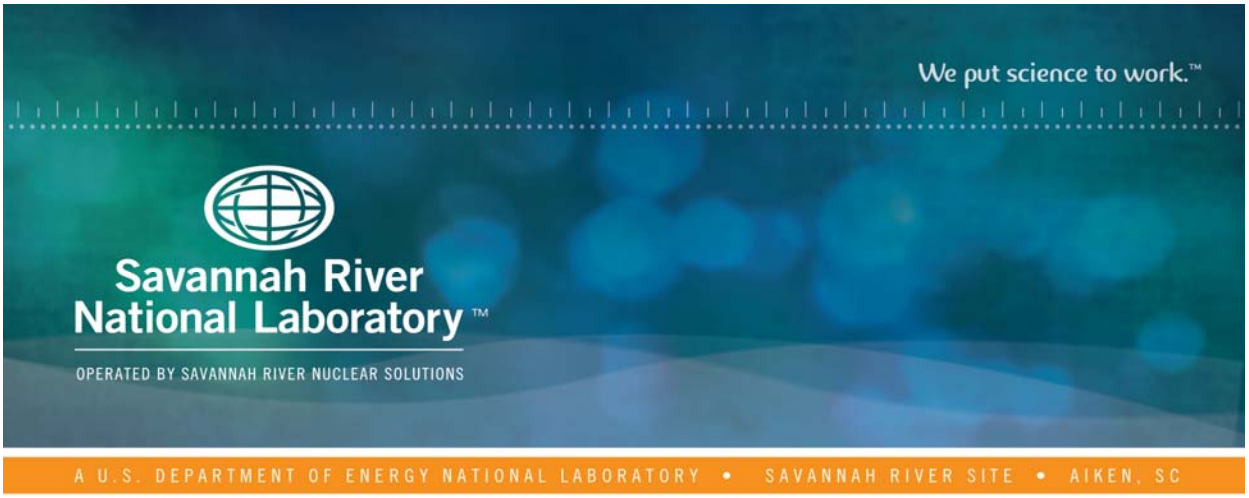

Gamma-Ray Imaging and Holdup Assays of 235-F PuFF Cells 1 & 2

T. J. Aucott

A. D. Brand

D. P. DiPrete

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REVIEWS AND APPROVALS

AUTHORS:

T. J. Aucott, Nuclear Measurements Date

A. D. Brand, Nuclear Measurements Date

D. P. DiPrete, Nuclear Measurements Date

TECHNICAL REVIEW:

C. DiPrete, Nuclear Measurements Date

K. M. Hammerton, Nuclear Measurements Date

J. C. Musall, SW&F-Area Engineering Date

APPROVAL:

R. H. Young, Manager Date
Nuclear Measurements

J. A. Hasty, 235-F Risk Reduction Project Management Date

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LIST OF ABBREVIATIONS

DSA	Digital Signal Analyzer
FRAM	Fixed energy, Response function Analysis with Multiple efficiencies
GeGI	Germanium Gamma-ray Imager
HEPA	High Efficiency Particulate Air (filter)
HPGe	High-Purity Germanium
ISOCS	*In Situ* Object Counting System
keV	kilo-electron-Volt
MCA	Multichannel Analyzer
MCNP	Monte Carlo N-Particle
MDA	Minimum Detectable Activity
NDA	Non-destructive Assay
PuFF	Plutonium Fuel Form (facility)
SRNL	Savannah River National Laboratory

1.0 Introduction

The Plutonium Fuel Form (PuFF) Facility, located in Building 235-F, was used to produce spheres and pellets of plutonium-238 for radioisotope thermal generators. Between 1978 and 1984, the PuFF Facility processed approximately 165 kilograms of Pu-238. The fuel was processed in nine shielded cells, organized into lines on the east and west side of the control room. Several wing cabinets on the east cell line were also used for processing.

When the facility was placed in standby in 1984, the shielded cells were not cleaned out of any remaining plutonium which might have been held up in the process. A large number of non-destructive assay (NDA) measurements were performed in 2014 from underneath the cells. These assays indicated approximately 240 grams of Pu-238 remained in the shielded cells, primarily in cells 1 and 2. Figure 1 below shows a reconstruction of the 2014 measurements overlaid on drawings of cells 1 and 2.

Savannah River National Laboratory (SRNL) Nuclear Measurements (L4120) was tasked with performing enhanced characterization of the holdup in the PuFF shielded cells. Assays were performed in accordance with L16.1-ADS-2460 using two high-resolution gamma-ray detectors. The first detector, an *In Situ* Object Counting System (ISOCS)-characterized detector, was used in conjunction with the ISOCS Geometry Composer software to quantify grams of holdup. The second detector, a Germanium Gamma-ray Imager (GeGI), was used to visualize the location and relative intensity of the holdup in the cells.

Carts and collimators were specially designed to perform optimum assays of the cells. Thick, pencil-beam tungsten collimators were fabricated to allow for extremely precise targeting of items of interest inside the cells. Carts were designed with a wide range of motion to position and align the detectors. A total of 24 measurements were made, each typically 24 hours or longer to provide sufficient statistical precision.

This report presents the results of the enhanced characterization for cells 1 and 2. The measured gram values agree very well with results from the 2014 study. In addition, images were created using both the 2014 data and the new GeGI data. The GeGI images of the cells walls reveal significant Pu-238 holdup on the surface of the walls in cells 1 and 2. Additionally, holdup is visible in the two pass-throughs from cell 1 to the wing cabinets.

This report documents the final element (exterior measurements coupled with gamma-ray imaging and modeling) of the enhanced characterization of cells 1-5 (East Cell Line).

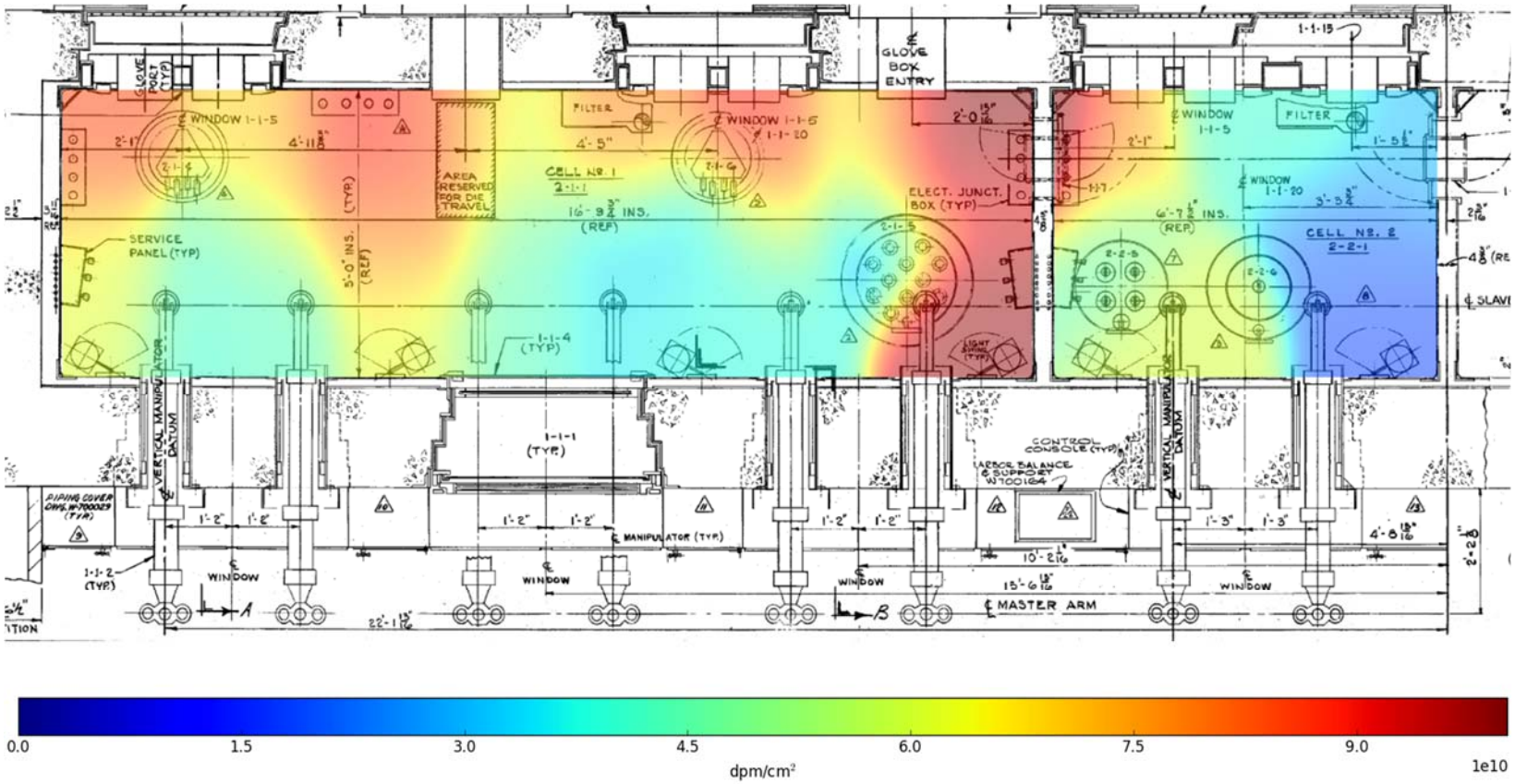

Figure 1: Heatmap showing measured floor activity in cells 1 and 2. Data is from the 2014 assays from underneath the cells. Units are 10^{10} dpm/cm^2.

2.0 Measurements

2.1 Equipment Used

2.1.1 In Situ Object Counting System (ISOCS) Detector

The ISOCS detector is a standard coaxial high-purity germanium (HPGe) detector with a relative efficiency of about 30%. This detector is "ISOCS-characterized" by Canberra, which means it was extensively calibrated at the factory and characterized using the Monte Carlo N-Particle (MCNP) transport code. The benefit of these detectors is that an assay configuration can be described in the ISOCS software, and a detector-specific calibration can be produced quickly.

The detector was placed in a matching 1" lead shield, which is also characterized in the calibration software. The shield was mounted to a pivoting assembly, allowing for adjustment of the detector pitch. The assembly was then bolted to a cart with a hydraulic lift. The resulting assembly allowed for a large amount of flexibility in positioning and alignment. A small laser pointer and laser range finder were also employed to aid alignment and to measure distances between the detector and locations inside the shielded cells.

Two different collimators were used with the ISOCS detector, both shown in Figure 2. The first was a wide-angle lead collimator with an opening of 90 degrees. This collimator was used to verify the total quantity of Pu-238 on the floor of the cells. This collimator is also ISOCS-characterized, and has been used extensively in the past to provide holdup measurements.

The second collimator is a pencil-beam tungsten collimator with a diameter of 4.23mm, which sits inside the lead shield. This collimator was custom made to provide a very narrow opening angle and consists of two pieces so that the thickness can be varied. The dimensions were chosen to target the high efficiency particulate air (HEPA) filters on the rear wall of the cells, approximately 2.2 meters away, without interference from other strong sources. Both collimators were modeled using the ISOCS software and were benchmarked to a calibration measurement using a NIST-traceable Ho-166m standard.

Data acquisition was performed with a digital signal analyzer (model DSA-LX), a portable multichannel analyzer (MCA) which provides high voltage and communications with a personal computer. Personnel controlled the data acquisition through the Genie 2000 software on a laptop.

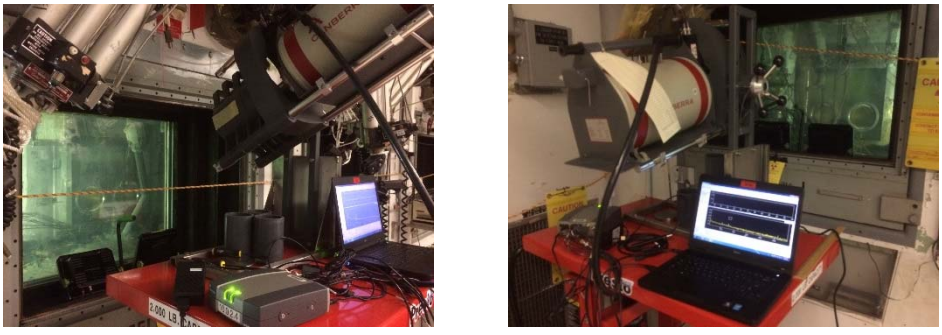

Figure 2: ISOCS detector in front of cell 1. Left: Employing the 90-degree collimator and aimed at the floor of the cell. Right: Employing the pencil-beam collimator and aimed at the back wall of the cell.

2.1.2 Germanium Gamma-Ray Imager (GeGI) Detector

The GeGI is a gamma-ray imaging detector which can create images using two different techniques: pinhole imaging and Compton imaging. The detector is a planar, double-sided strip readout HPGe detector. The configuration allows for the location of gamma-ray interactions to be determined precisely within the crystal. The interaction positions are then used to recreate the distribution of radioactivity in the field of view. The GeGI also includes an ordinary digital camera, which is used to map the measured distribution onto a photograph.

For cells 1 and 2, the imager was used in pinhole mode to obtain better imaging resolution. In this mode, a thick lead shield with a 60-degree pinhole opening is placed in front of the detector. This method greatly improves the imaging resolution due to the small opening. First, the field of view is limited to 60 degrees in front of the detector. Second, this technique works best for low-energy photons, so only the 99 keV and 153 keV emissions of Pu-238 are useful. Finally, the efficiency is greatly reduced due to the lead shielding (although this tended to be less of a problem due to the high dose in front of cells 1 & 2).

The GeGI was placed on the custom cart, shown in Figure 3, which was built for assays of the PuFF cells. The cart includes a mount which allows for adjustment of both the pitch and yaw of the GeGI. The detector was aimed at both the floor and the wall of the cells and was moved as necessary to capture the entire visible area of both cells. Data acquisition and analysis are performed by a tablet which is attached to the detector. All analysis is done by the device software.

The GeGI results were used for two purposes. The first was to verify the distribution of holdup on the floor of the cells, as observed in the 2014 assays. The second was to determine the extent of holdup on the HEPA filters and cell walls. The filters and other contaminated objects on the walls were measured explicitly with the ISOCS detector to determine quantities of holdup, and the GeGI was used to extend these results to the entire wall.

Figure 3: GeGI (on the right) and ISOCS (on the left) detectors in front of cell 1 windows.

2.2 Measurement Approach

Measurements were made at the plane where the outer-most glass pane sat prior to being removed. Measurement live time ranged from 5 minutes for the wide-angle ISOCS shots, up to 100 hours on the relatively clean sections of the wall. Most of the measurements were about 24 hours long.

Background measurements were not made for these assays, as the locations of interest have much higher doses than any nearby location. Any background measurements would have inevitably measured some of the material of interest, resulting in an assay value which was biased low. Background was minimized by using 1" thick lead shielding on both detectors. The ISOCS shielding extended behind the detector as well as in front.

Wide-angle measurements of the floor were made with a 0.79 mm sheet of cadmium in front of the detector. The cadmium sheet was used to attenuate low-energy photons which would otherwise overwhelm the detector. This cadmium was included in all models.

Data was acquired with 16,000 channels over a range of 3000 keV, with a resulting 0.18 keV/channel. An example spectrum is shown in Figure 4. The primary Pu-238 peaks of interest are found at 99 keV, 153 keV, and 766 keV. Spectra were saved in Genie and analyzed using PeakEasy.

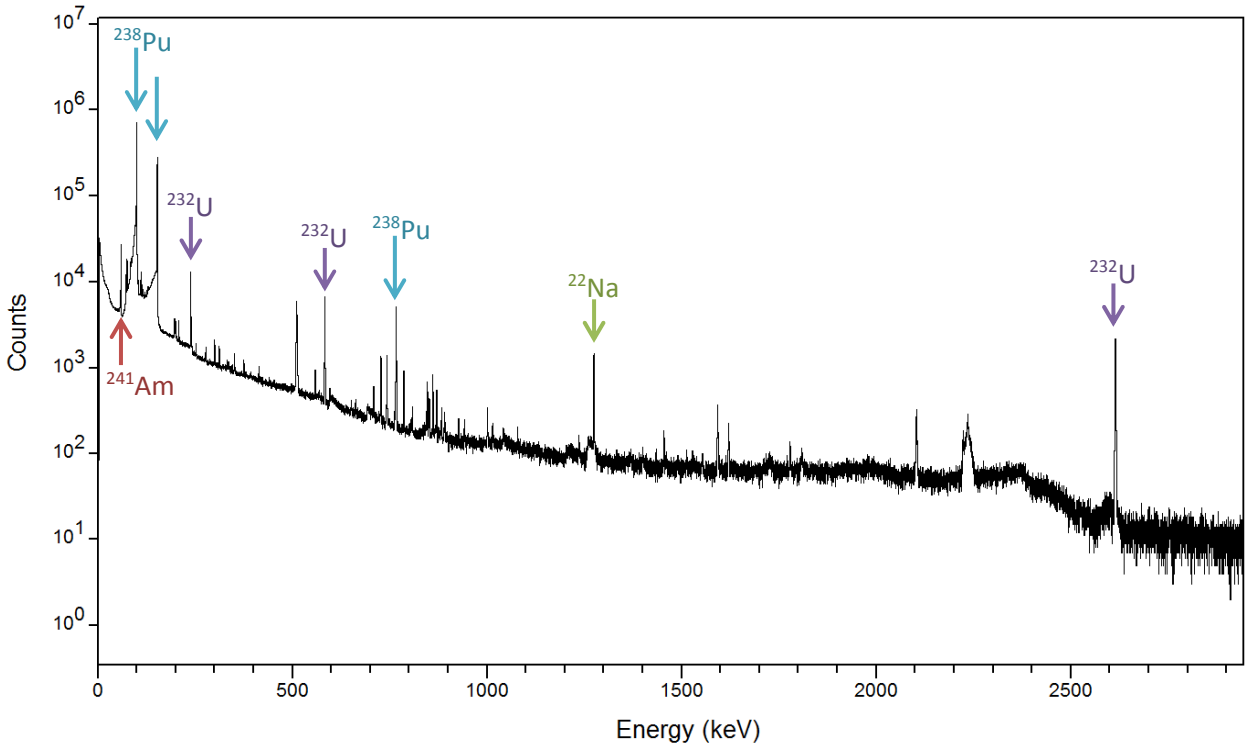

Figure 4: Example energy spectrum acquired by the ISOCS detector, showing the primary peaks of interest. This spectrum is the sum of the four wide-angle ISOCS measurements, totaling 1500 seconds of live time.

3.0 Analysis

3.1 Calibration

ISOCS models were built for each of the measurement acquisitions. The targeted area of interest was assumed to be uniformly covered with Pu-238. For small objects, such as the HEPA filters, this is a reasonable assumption. For the floor of the cells, this was compensated for by making multiple measurements at different locations. An example rendering of the model used for the floor of cell 1 is shown in Figure 5.

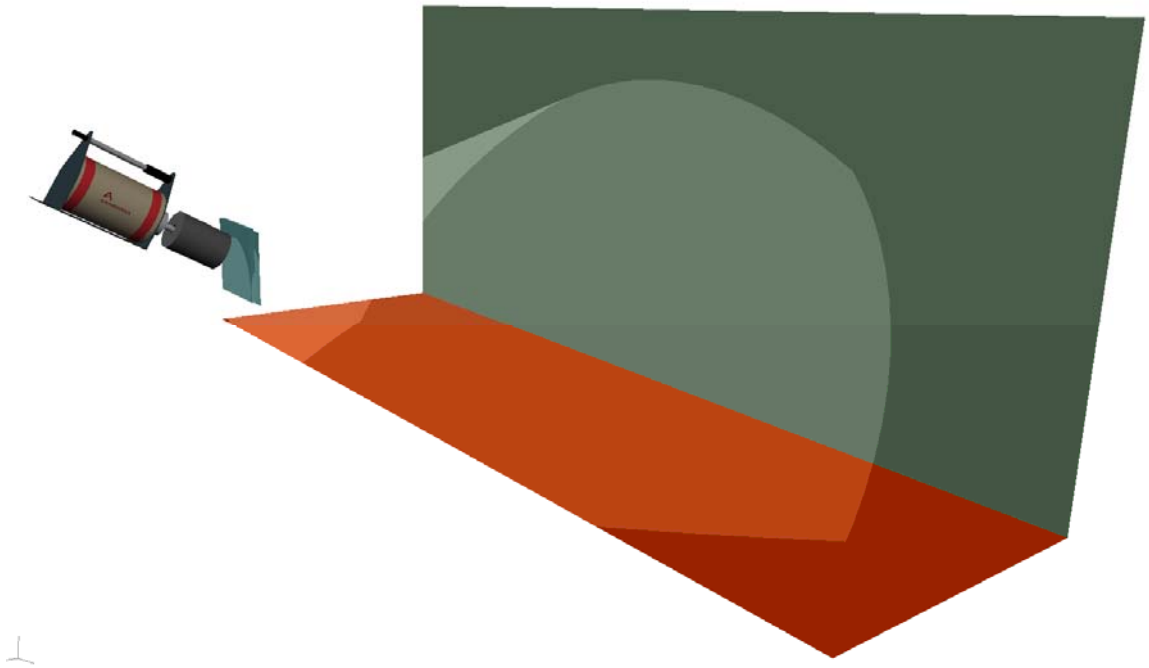

Figure 5: Example ISOCS model, showing the ISOCS detector looking through the central window of cell 1 towards the floor of the cell. The orange area indicates that the entire floor is assumed to be uniformly contaminated in the model.

The two collimator configurations were benchmarked to a NIST-traceable Ho-166m source. The source was measured first with no collimator to determine any bias in the ISOCS model. This measurement is made regularly to account for any dead layer growth on the surface of the germanium crystal. The result is an energy-dependent bias correction which is applied to all the ISOCS results.

Next, both collimator configurations were measured with the same source, and the collimators were modeled in ISOCS. The lead collimator was already included in the software and characterized by default. Since the tungsten collimator was custom-built, it was modeled explicitly in the software.

The ISOCS efficiencies are compared to the measured efficiencies to ensure that ISOCS can correctly model the effects of the collimators. Results are shown in Figure 6 and Figure 7. The 90-degree lead collimator model matched to within 3% of the measured values, well within the estimated ISOCS uncertainty of about 10%. The tungsten collimator model matched well given the large measurement uncertainties involved; all measurements matched to within 2-sigma.

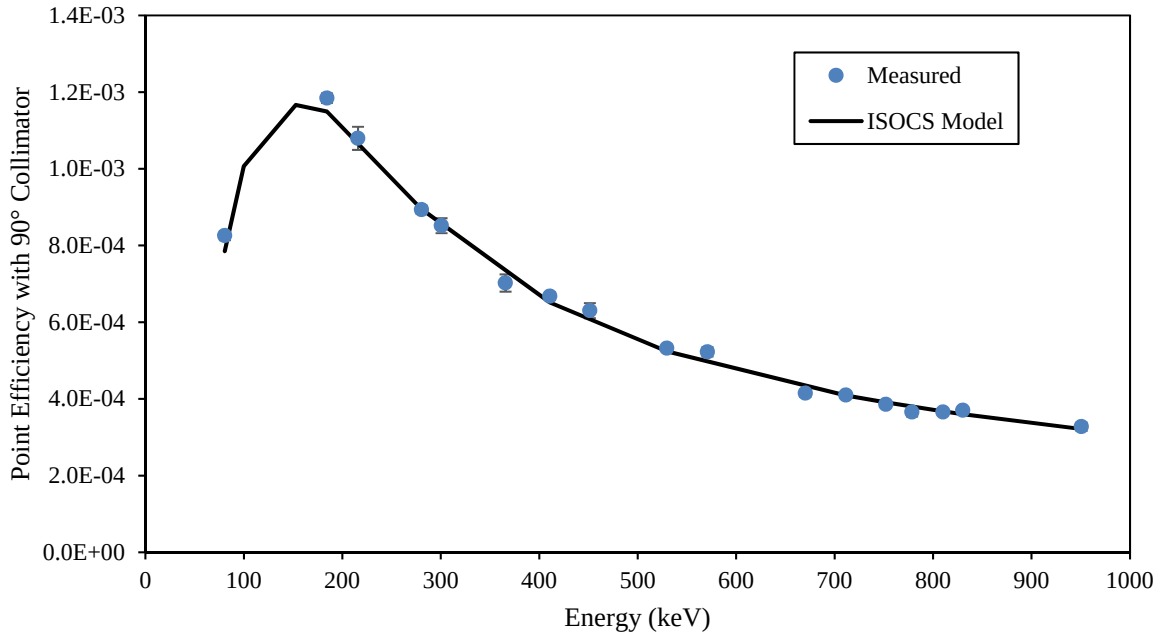

Figure 6: Benchmark of measured efficiency, using a Ho-166m source, with the ISOCS modeled efficiency. This configuration is with the 90-degree lead collimator.

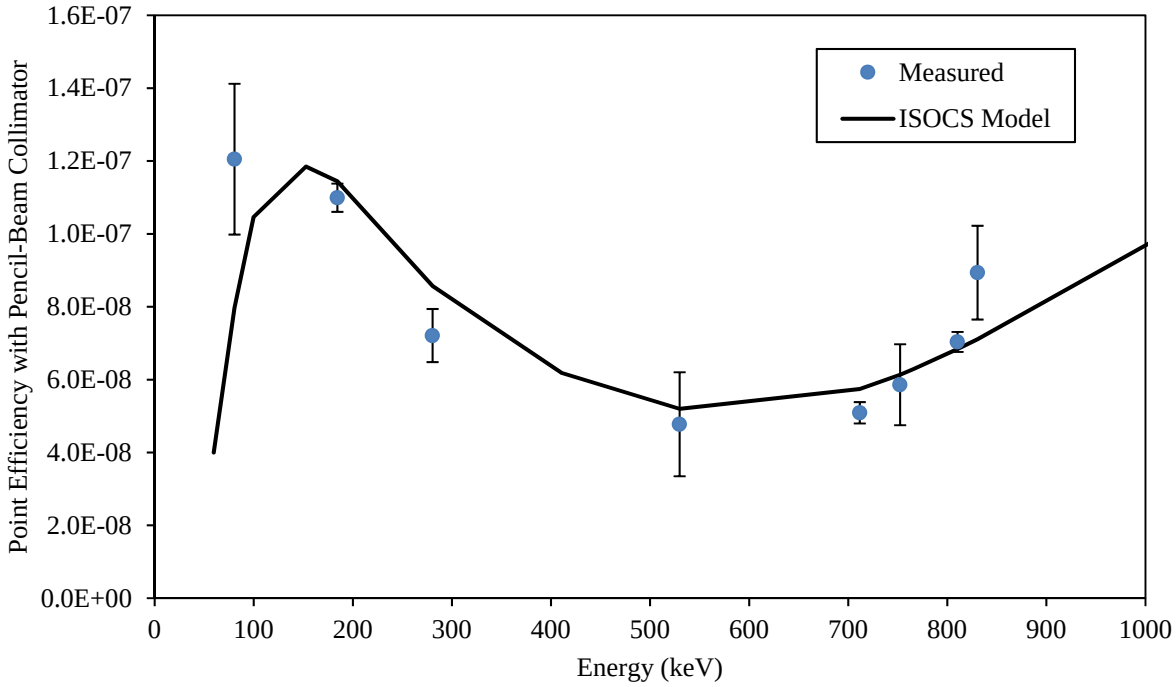

Figure 7: Benchmark of measured efficiency, using a Ho-166m source, with the ISOCS modeled efficiency. This configuration is with the full thickness tungsten collimator in place.

3.2 Attenuation Correction

The floors of the PuFF cells tend to be cluttered with a variety of machinery used in the fuel form process. It is difficult to know a priori whether or not plutonium is behind a thick piece of machinery which would shield any gamma rays. To address this, one or more additional layers of attenuating material can be added to the model. For this analysis, the intervening material is assumed to be stainless steel.

The Pu-238 mass m_i is first calculated for each of the three major gamma rays: 99, 153, and 766 keV, where the subscript *i* indicates which of the three gamma rays is used. Generally, the 99 keV results are biased lower than the others, due to its greater attenuation. This indicates that there is some steel of unknown thickness *x* which has not been included in the model. The Pu-238 mass is then recalculated with the additional steel:

$$m'_i = m_i \cdot \exp(\mu_i \cdot x)$$

where the attenuation coefficient μ_i for stainless steel is known for each gamma ray. The thickness *x* is then varied until the difference in masses m'_i is minimized by least-squares. The estimated thicknesses are about 1.1 cm of steel in cell 1 and 0.7 cm of steel in cell 2.

It was noted that even with this correction, the middle energy (153 keV) gave a Pu-238 mass which was consistently lower than the other two gamma rays. The same phenomenon was observed in the 2014 assays from underneath the cell floors. This suggests that the model of a single attenuating layer does not perfectly describe the material and shielding distribution in the cells.

The steel can be re-estimated, using the assumption that there are two material distributions, one unshielded and one shielded by stainless steel. This increases the estimated steel thicknesses, as well as the total amount of holdup. Based on the 2014 assay results, it is believed that the shielded activity is due to material in the furnaces (for cell 1) and the coolers (for cell 2).

The measurements of the HEPA filters and the walls were not corrected for attenuation. In general, the two lower energy lines matched well for the filter and wall measurements, with no additional adjustments. The 766 keV line was consistently higher than the other gamma rays – sometimes by an order of magnitude. This is likely due to gamma rays making it through the thick shielding. An inch of lead, which was the thickness of the outer shielding for all configurations, still allows 8% of these gamma rays through. The 766 keV line was ignored when analyzing the data targeting the filters and walls.

3.3 Isotopic Identification

The measurements through the windows of cells 1 and 2 provided a good opportunity to make a precise isotopic measurement of the material inside. Although not critical to the determination of the holdup, it provides good confirmation that there are no unexpected isotopes inside the PuFF cells.

The wide-angle measurements were summed together and analyzed using the Fixed energy, Response function Analysis with Multiple efficiencies (FRAM) software. A custom parameter file was built to analyze Pu-238 (as FRAM is typically used to analyze material containing primarily Pu-239). The analysis provides relative quantities of each plutonium isotope, as well as other actinides such as Am-241, Np-237, and U-232.

4.0 Results

4.1 Total Holdup Results

The ISOCS measurements are first analyzed with the appropriate calibration for the floors and the HEPA filters. Floor measurements are then corrected using the attenuation analysis described in section 3.2. Results for the floor are in good agreement with previous assays, as shown in Table 1. As observed previously, the measurements show excess 766 keV photons, suggesting a second, highly-attenuated source. Based on the assay results from 2014, it is believed that the activity difference between these estimates is due to material in the furnaces (for cell 1) and the coolers (for cell 2). In Table 1, the furnaces and coolers are grouped together for each cell, as this assay did not determine the exact location.

Table 1: Summary of total Pu-238 holdup measured in cells 1 and 2. The results from the 2014 assay are shown on the right for reference. Uncertainties are stated at one-sigma. When tabulating totals, MDA values are included as 0 ± *MDA*.

Cell 1	**Pu-238 (g) 2017**	**Pu-238 (g) 2014**
Floor	91.5 ±12%	114 ±5%
Walls	41.5 ±22%	N/A
HEPA	2.1 ±10%	N/A
North furnace	60.1 ±21%	17 ±31%
South furnace		43 ±28%
Cooler		10.7 (MDA)
Die pass-through	3.3 ±22%	N/A
Glovebox entry	7.7 ±22%	N/A
Cell 2	**Pu-238 (g) 2017**	**Pu-238 (g) 2014**
Floor	34.9 ±12%	36 ±9%
Walls	16.3 ±22%	N/A
HEPA	0.7 ±10%	N/A
North cooler	16.9 ±23%	11.9 ±11%
South cooler		12.7 (MDA)
Cell 1 Total	**206.1 ±17%**	**174 ±17%**
Cell 2 Total	**68.8 ±17%**	**47.9 ±23%**

HEPA filters were measured twice, once with the full-thickness pencil-beam collimator, and again with a half-thickness collimator. The full-thickness measurement was designed to view just the HEPA filter, while the half-thickness measurement was performed to ensure that the edges of the filter were not missed. The half-thickness results for cell 2 are significantly higher, but this is consistent with the GeGI images (shown in Figure 9), which show significant material on the wall surrounding the HEPA filter, rather than on the filter itself.

The cell walls, and two other locations of interest in cell 1 (the die pass-through and glovebox entry), are analyzed using the GeGI image, as described in section 4.4.

4.2 Plutonium Isotopics

Table 2 shows the measured plutonium isotopics, as determined by the wide-area measurements for both cells 1 and 2. The results are consistent with typical heat-grade plutonium, which initially contains above 80% Pu-238. The observed Pu-238 content was 74.8%, which would be expected after a few decades of decay. This table also shows other nuclides identified in these cells, reported relative to total plutonium. Am-241, Np-237, and U-234 values are also consistent with decades of radioactive decay. In particular, the Am-241 / Pu-241 ratio is a good indicator of age. This ratio dates the material at 33 years old (± 1 year), consistent with the last year of operation of the PuFF facility.

Table 2: Measured plutonium isotopics for material in cells 1 and 2. Data is averaged across the four wide-area ISOCS measurements. Uncertainties are stated at one-sigma.

Isotope	**Weight %**
Pu-238	74.82 ± 0.7%
Pu-239	16.58 ± 2.8%
Pu-240	7.31 ± 5.6%
Pu-241	0.09 ± 2.9%
Pu-242	1.20 (calc)*
Nuclide	**g / gPu**
Am-241	3.55E-3 ± 6.9%
Np-237	9.85E-4 ± 3.7%
U-232	4.07E-7 ± 0.9%
U-234	3.21E-1 ± 7.3%

* Pu-242 is not measured directly by gamma; it is calculated by FRAM.

4.3 GeGI Imaging

The GeGI is used to create multiple pinhole images at various locations. The 99 keV photon is the strongest emission from Pu-238, and this was the primary gamma ray used to make the pinhole images. Images made from 153 keV (Pu-238) and 59 keV (Am-241) are very similar, but have less precision due to the lower count rates. The images include only gamma rays which interacted in the front of the detector, due to the low penetration of these low-energy photons into the detector crystal.

Each GeGI image was overlaid on a camera image taken during acquisition. The pinhole image was projected onto a plane at 2.2 meters from the detectors, corresponding to the back wall of the cells. Figure 8 and Figure 9 show compilations of the multiple images made for each cell. Each red circle shows an individual image, and each image covers an area about 2.4 meters wide.

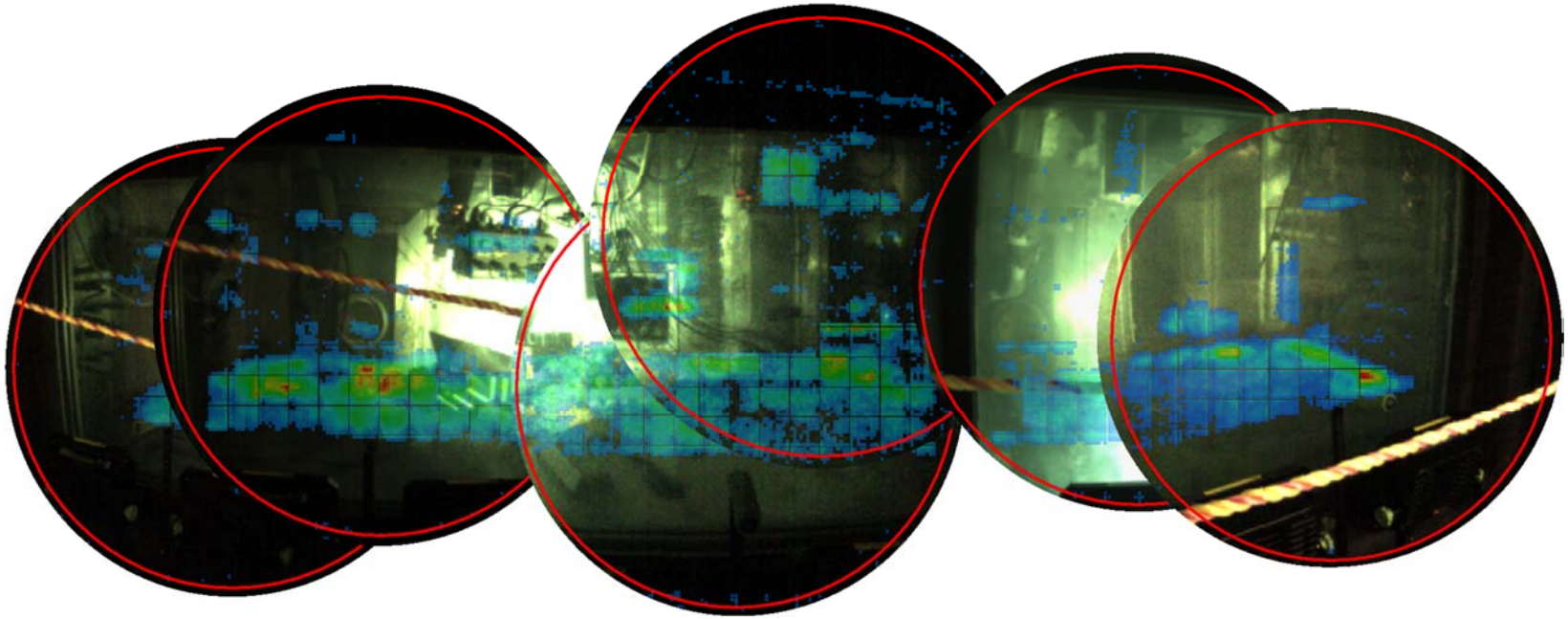

Figure 8: Compilation of six pinhole images of cell 1. The gamma ray imaged here is the 99 keV photon from Pu-238 only.

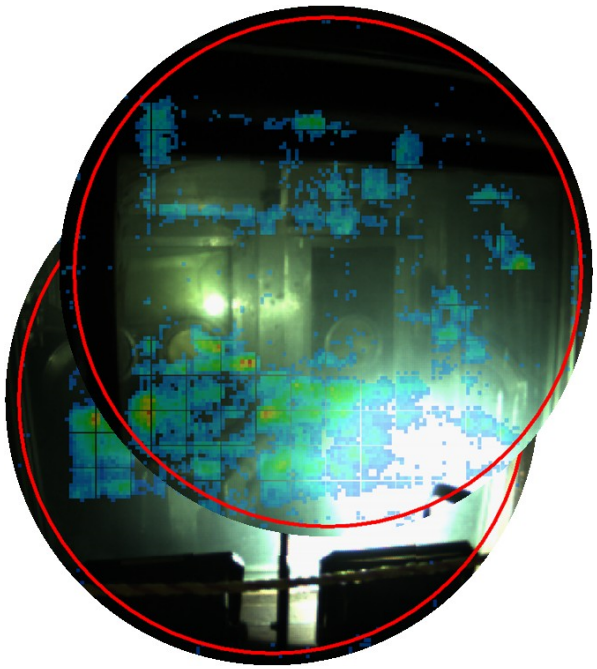

Figure 9: Compilation of two pinhole images of cell 2. The gamma ray imaged here is the 99 keV photon from Pu-238 only.

4.4 Wall Holdup Results

The walls of the cells pose an interesting challenge. The activity is difficult to quantify with a traditional assay, because the floor of the cell will overwhelm the small signal from the wall contamination. The custom collimator used here works well for discrete objects, such as the HEPA filter, but is unable to quantify a wide area of wall with non-uniform contamination.

To estimate the contamination on the wall, results from both the GeGI and ISOCS assays are combined. First, the ISOCS results from the HEPA filters are used as reference points in each cell, to essentially "calibrate" the images from the GeGI. The coarse pixels in the GeGI image are each 17 cm wide at the surface of the wall, so the HEPA filter (~32 cm wide) fills four full pixels.

Next, the GeGI results are analyzed using the vendor-supplied software to obtain the total grams on the visible portions of the wall. Using the known size of the pixels on the surface of the wall, the surface contamination (g/cm2) is calculated from the analysis software for each of the walls. The results from this analysis are shown in Table 3. This table also includes two additional locations (the die pass-through and glovebox entry) which are observed to be contaminated.

Table 3: GeGI results for holdup on cell walls and other measured surfaces. Surface areas are taken from engineering drawings.

Cell 1	Surface area (m^2)	GeGI coverage	Contam. (g/m^2)	Pu-238 (g)
Rear wall	9.871	61%	2.92	28.8
Left wall	3.174	31%	0.85	2.7
Right wall	3.174	48%	3.14	10.0
Die pass-through	--	100%	--	3.31
Glovebox entry	--	100%	--	7.66
Cell 2	**Surface area (m^2)**	**GeGI coverage**	**Contam. (g/m^2)**	**Pu-238 (g)**
Rear wall	4.091	48%	1.96	8.0
Left wall	3.174	0%	1.96	6.2
Right wall	3.174	69%	0.64	2.0

Most walls were between 1/3 and 2/3 visible, except for the left wall of cell 2. The concrete support in the middle of the control room prevented the detector from being aimed at this wall. For conservatism, it is assigned the same contamination as the rear wall. Additionally, the non-visible portions of each wall (typically the top third) are assigned the same contamination as the rest of the wall. This is conservative, as it is more likely that the top of each wall is less contaminated than the bottom.

Uncertainties for these results are difficult to assign, as the details of the vendor software for the GeGI are obscured. An estimated 20% holdup uncertainty are assigned to these values, in addition to the measurement of the HEPA filter by the ISOCS detector, for a total uncertainty of 22% (one-sigma).

In addition to the GeGI analysis, multiple measurements were made of the back walls using the ISOCS detector and pencil-beam collimator. Clean locations and contaminated equipment were targeted to understand the range of contamination on the walls. However, the results were not as accurate as the GeGI image, as it was difficult to target only a small, clean area of the wall. The cleanest location was found in cell 1, directly below the HEPA filter, with a surface contamination of 0.09 g/m^2 (± 15%).

There is no indication from the GeGI or ISOCS data that the window interiors are contaminated, as measured and imaged activity was correlated with objects on the walls, rather than the rectangular shape of the window pane. If the windows were contaminated, this material would be three times closer to the detectors, meaning that any measured activity would be lower by a factor of nine. Thus, even if the clean location mentioned above were entirely due to the window, this would only be a surface activity of 0.01 g/m^2, resulting in less than 0.05 g for all five windows. This quantity is well below the precision of the results reported here.

5.0 Conclusions

Multiple gamma-ray assays were performed to quantify and image the holdup in PuFF cells 1 and 2 in 235-F. These measurements extended the previous assays by including the walls and HEPA filter, as well as using a state-of-the-art gamma-ray imager to observe the distribution of material.

Measured plutonium quantities on the floor of the cells matched well with past assays. Although the coolers and furnaces were not measured explicitly, the observed rate of high-energy and low-energy photons was consistent with the past measurements of these locations.

Total measured Pu-238 content was measured to be 206.1 grams (±17%) for cell 1, and 68.8 grams (±17%) for cell 2. These assay results increase the holdup values by 32 grams (cell 1) and 21 grams (cell 2). This increase, however, is due almost entirely to the inclusion of the walls, which was not considered in the past.

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