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High Purity Germanium γ-PHA Assay of Uranium Scrap Cans Used in 321-M Facility

Saleem R. Salaymeh, Raymond A. Dewberry, and Vito Casella

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ABSTRACT

The Analytical Development Section of SRTC was requested by the Facilities Disposition Division (FDD) to determine the holdup of enriched uranium in the 321-M facility as part of an overall deactivation project of the facility. The 321-M facility was used to fabricate enriched uranium fuel assemblies, lithium-aluminum target tubes, neptunium assemblies, and miscellaneous components for the production reactors. The facility also includes the 324-M storage building and the passageway connecting it to 321-M. The results of the holdup assays are essential for determining compliance with the Solid Waste's Waste Acceptance Criteria, Material Control & Accountability, and to meet criticality safety controls. This report describes and documents the use of a portable HPGe detector and EG&G DART® system that contains a high voltage power supply, signal processing electronics, a personal computer with Gamma-Vision software, and space to store and manipulate multiple 4096-channel γ-ray spectra to assay for ^{235}U content. The system was used to assay a large number of scrap cans used to store highly enriched uranium (HEU) chips and filings. This report includes a description of two efficiency calibration configurations and also the results of the assay. A description of the quality control checks is included as well.

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High Purity Germanium γ-PHA Assay of Uranium Scrap Cans Used in 321-M Facility

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1. INTRODUCTION

The 321-M facility was used to fabricate highly enriched uranium (HEU) fuel assemblies, lithium-aluminum target tubes, neptunium assemblies, and miscellaneous components for the production reactors. The facility also includes the 324-M storage building and the passageway connecting it to 321-M. The facility operated for 25 years. During this time thousands of uranium-aluminum-alloy (U-Al) fuel tubes were produced. After the facility ceased operations in 1995, all of the easily accessible U-Al was removed from the building, and only residual amounts remained. The bulk of this residue is located in the equipment that generated and handled small U-Al particles and in the exhaust systems for this equipment (e.g., chip compactor, casting furnaces, log saw, lathes A & B, cyclone separator, Freon™ cart, riser crusher, …etc).[1] In addition to these examples there are 268 pigs that were used for storage and shipping of HEU.

Facilities Disposition Division (FDD) has requested technical assistance from the Analytical Development Section (ADS) of the Savannah River Technology Center to determine the holdup of enriched uranium in the 321-M facility as part of an overall deactivation project of the facility.[2] This project includes the dismantling and removal of all HEU to the extent practical. ADS was tasked to conduct holdup assays and waste residue assays of all components removed from the facility. A large number of scrap cans was used by the facility to store highly enriched uranium chips and filings for reprocessing. The scrap cans were designed to be critically safe, which made them extremely useful during the deactivation of the facility. These cans provided a geometrically safe container for placement of the residue, filings, chips, and sweepings of HEU remaining in the building. Since the assays are important for material control and accountability (MC&A), criticality safety, and waste management purposes, it was important to obtain the best gram value possible. The method of choice for determining the HEU content in each scrap can is by using nondestructive assay (NDA).

To accomplish this, we set up an assay station in 324-M that consisted of a turntable and portable HPGe gamma pulse height analysis system (γ-PHA). The 324-M assay station was especially suited to obtain a transmission-corrected assay of each of these fixed geometry scrap cans. This report discusses the detector efficiency calibration and use of the portable HPGe detection system[3] to obtain an assay of ^{235}U content in each scrap can.

2. EXPERIMENTAL

A photograph of the assay station that we assembled in Building 324-M is shown in Figure 1. Figure 2 shows a closer view of the components used for the data acquisition. In Figure 2 it is clear that the assay station was assembled so that the portable HPGe detector views the sample scrap can in a far field configuration. At the 324-M station the scrap can stands on a turntable of fixed height inside of a contamination area (CA). The portable detector was adjusted to the optimum height so that it views the scrap can with greatest efficiency from outside of the CA. The sample to detector distance is fixed at 48 inches. Behind the scrap can turntable shown in both Figures 1 and 2 is the transmission source stand. The stand is set so that the removable source of 4.41 g U-235 will be at a fixed source to detector distance of 63 inches and so that photons from decay of it must pass through the sample scrap can to interact with the detector.

The photograph in Figure 1 shows three personal computers (PC) used at the assay station. The acquisition and analysis system consists of the portable HPGe detector, an EG&G DART® electronics package shown on the extreme left of the assay table, and the PC immediately to the right of the DART® electronics box. The DART® system contains the high voltage power supply and the signal processing electronics. The PC contains the Gamma-Vision software that converts it to a full function multi-channel analyzer for data acquisition and analysis.[3]

Figure 1. A photograph of the assay station in Building 324-M.

The PC in the center of the assay table in Figure 1 contains a Microsoft® excel spreadsheet that we wrote to allow us to provide on the spot results for the transmission corrected far field assays. The spreadsheet is constructed so the input data of counting

time and peak area in the 185.70 KeV region of interest in the sample γ-ray spectrum and sample transmission spectrum instantly yield the transmission correction value as well as assay results and uncertainty for each can. The third PC on the right of the table in Figure 1 contains a Deming least squares quadratic curve that provides us with a second assay result based on a stored transmission correction curve.[4] We provide further details of the Microsoft® excel spreadsheet and of the Deming curve in the next section. All of the acquisition, analysis, spreadsheet calculations, and curve fitting software could have been run on a single PC, but having three available eliminated the requirement of continually moving the software around the desktop.

Figure 2. A closer view of the components used for the data acquisition.

A sample assay consists of performing two γ-PHA acquisitions with each can. One acquisition is performed on the scrap can alone and a second is performed with the transmission source placed behind the scrap can on the fixed stand. The second acquisition then contains sample events and transmission source events in the 185 KeV γ-ray region of interest. The transmission attribute of each sample can is calculated from

$$\mathrm{T(can)} = \frac{\mathrm{cps(canT)} - \mathrm{cps(can)}}{\mathrm{cps(source)}}, \qquad (1)$$

where cps(canT) is the sample plus transmission source rate, cps(can) is the can-only rate, and cps(source) is the source-only rate. The source-only rate is obtained with each shift of acquisitions, and serves also as a quality control (QC) check for each shift of analyses.

For the QC check, the 4.41-g source is counted at a distance of 63 inches and is analyzed by equation (2).

$$^{235}\mathrm{U} = (\mathrm{K_p})(\mathrm{cps})(\mathrm{d})^2 = (2.36\times10^{-5})(\mathrm{cps})(63\times2.54)^2, \quad (2)$$

where the first term is the point source calibration constant in units of g-sec/cm^2, and d is the acquisition distance in units of cm. Note in Table 2 the T0 acquisition of 7/6/00 had a detection rate of 2210/300 = 7.367, which yields a measured result of 4.45 g for the source. This is in extremely good agreement with the known content of 4.41 g. In subsequent QC checks we required the T0 QC check to have a detection rate of 7.30±0.73 cps.

The measured contents of each can are calculated using the transmission-corrected point source configuration of equation (3).

$$^{235}\mathrm{U} = (\mathrm{K_p})(\mathrm{cps})(\mathrm{d})^2(\mathrm{Cf}) = (2.36\times10^{-5})(\mathrm{cps})(48\times2.54)^2(\mathrm{Cf}), \quad (3)$$

where the acquisition distance is 48 inches, and Cf is the transmission correction factor for each can determined from the results of equation (1). For each can Cf is taken to be $\sqrt{\mathrm{T}}$, where T is determined from the measured data using (1).

For each can of material, the transmission attribute is a combination of absorption of the 185 KeV γ-ray by the container and by the ^{235}U residue inside. Therefore every measured correction factor must be at least as large as that of the container (can) alone. In order to determine the can-only transmission, we acquired the data listed in Table 1. These data represent can-only data using several M-Area sources of solid HEU.

Table 1 lists seventeen HPGe spectra acquired at the 324-M scrap can assay station in late April 2000. The first two rows and the fifth row represent background acquisitions, all of which have detection rates of the 185 KeV γ-ray in agreement with (0.115±0.014) cps. Figure 3 shows a background spectrum. For the rest of the treatment of the data of Table 1 we do not make the insignificant correction of subtraction of this very small background component.

The third and fourth acquisitions (WT2021At and WT2021Ac) represent traditional measures of the transmission of the 185 KeV γ-ray through a can that we have designated the standard scrap can. That is, WT2021At is a QC type spectrum acquired with source 2021a on the transmission source holder exactly as in the spectrum used in equation (2). The γ-ray spectrum for source 2021a is shown in Figure 4. The standard scrap can is shown in the photograph of Figure 5. Spectrum WT2021Ac was acquired with the standard scrap can placed between the source and the detector to measure the transmission through the standard scrap can. In the last column of Table 1 we have listed the transmission factor for the can alone, which is simply the ratio of the detection rate of acquisition WT2021At to that rate in WT2021Ac. That ratio is (7.623±0.122)/(3.563±0.087) = (2.14±0.28). (Henceforth for these Table 1 measurements we do not carry the uncertainty.) In the configuration of these two

acquisitions the transmission factor of 2.14 represents transmission of the 185 KeV photon through two sides of the standard can. For subsequent acquisitions of Table 1, the configuration represents transmission through only one side of the can, which is representative of how actual samples will be analyzed. Therefore the transmission correction factor is taken to be the $\sqrt{T}$ = 1.46 for the configuration of the third and fourth acquisitions of Table 1.

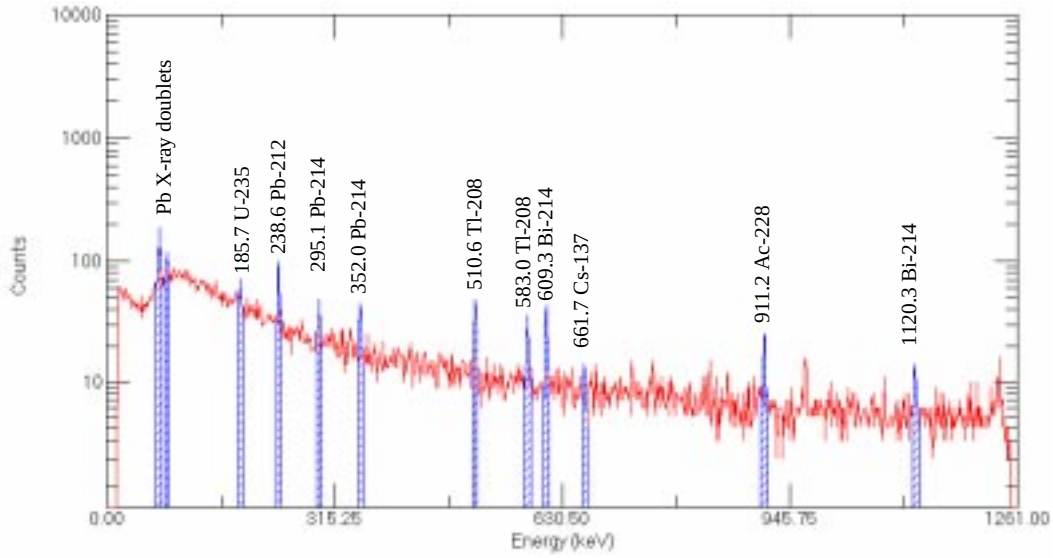

Figure 3. 324-M Background Spectrum.

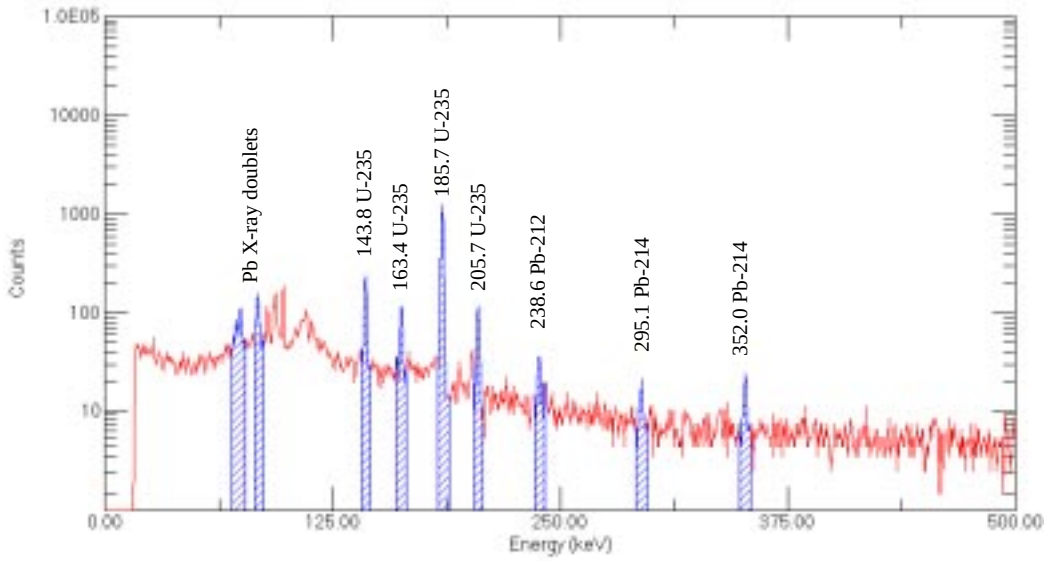

Figure 4. Standard 2021A Spectrum Acquired at the 324-M Assay Station.

The remaining twelve acquisitions of Table 1 all represent comparisons between spectra acquired from a standard source of ^{235}U inside the standard can with a spectrum acquired from the identical source and distance not inside the can. Thus the ratio of the detection rate of the 185 KeV photon from outside the can to that from inside the can represents an exact measure of the absorption of the photon by the can alone. Using six different sources of ^{235}U content ranging from 0.69 g to 98.31 g we obtained the transmission factors listed in the last column of Table 1. These transmission factors represent the transmission correction factor for one can thickness and range from 1.58 to 1.93. Overall we have seven measures of the can-only transmission correction factor that yield an average and standard deviation of 1.66±0.15. Any sample transmission correction factors measured below that range must be considered unrealistically low, and any significantly above that range must be regarded as a sample that contains particularly densely packed residue.

Table 1. List of data used to obtain empty scrap can transmission correction.

Spectrum Number	Description of Setup	185 KeV Area	Count Time (s)	CPS	T Corr.
BKG424-1	Bkg TT & T-source holder	331±51	2537	0.130	
BKG424-2	Bkg TT & T-source holder not in view of detec.	182±40	1800	0.101	
WT2021At	4.20g on T-source holder & TT w/o sample	4574±73	600	7.623	2.14
WT2021Ac	4.20g on T-source holder & TT with std can (5301) in front of source	2138±52	600	3.563	
std can	std can by itself rotating	57±27	600	0.095	
WT2023Aic	.69g inside the std can	932±31	600	1.553	1.62±0.07
WT2023Aoc	.69g centered outside the std can & TT (not rotating)	1506±40	600	2.51	
WT2021Aic	4.20g inside the std can	5538±78	600	9.23	1.58±0.03
WT2021Aoc	4.20g centered outside the std can & TT (not rotating)	8757±95	600	14.59	
WT2014Aic	1.01g inside the std can	1319±37	600	2.198	1.61±0.06
WT2014Aoc	1.01g centered outside the std can & TT (not rotating)	2128±48	600	3.546	
WT2016Aic	39.11g inside the std can	34370±200	600	57.283	1.65±0.01
WT2016Aoc	39.11g centered outside the std can & TT (not rotating)	56740±250	600	94.566	
WT2019Aic	15.67g inside the std can	17359±141	600	28.931	1.74±0.02
WT2019Aoc	15.67g centered outside the std can & TT (not rotating)	30151±180	600	50.251	
WT2026Aic	98.31g inside the std can	31708±193	300	105.693	1.93±0.01
WT2026Aoc	98.31g centered outside the std can & TT (not rotating)	61132±259	300	203.773	
Average Can Transmission Correction					**1.66**

Figure 5. A photo showing the scrap can that was used for calibration.

2.1 Transmission Correction Technique

In this report we use two methods of transmission correction for each sample analyzed. Our first method is what would generally be regarded as the traditional shine-through method widely used in γ-PHA nondestructive assay.[5] The second method involves an empirical correction using an interactive Deming least squares quadratic curve to assign an assay value. We describe that method in the next section.

The content of each scrap can is measured by the traditional transmission-corrected assay using a combination of data as described above and using equations (1) and (3). Each assay is shown completely in a single line of the spreadsheet of Table 2. The first two columns in Table 2 designate the acquisition date and the scrap can number. The third column denotes the count time for the sample plus source acquisition, and column four

lists the area under the 185 KeV peak for this count. Column five denotes the count time for the sample only (no transmission source), and column six denotes the area under the 185 KeV peak for that count. Column seven lists the transmission correction factor for the sample. This value is determined by $\sqrt{T}$, where T is calculated from equation (1). The mass of HEU in the can is then calculated by equation (3), and the uncertainty is determined from a propagation of the 2σ uncertainties in each of the multi-channel analyzer fits to the areas under the 185-KeV peaks. This uncertainty is propagated in equation (3) in both the (cps) term and in the (Cf_t) term.

The two sigma uncertainty for each far-field measurement is given by Equation (4):

$$2\sigma_u = M_u 2\times\sqrt{\left(\frac{\sigma_c}{C_c}\right)^2+\left(\frac{\sigma_T}{C_T}\right)^2+\left(\frac{7}{236}\right)^2+(0.0112)^2} \qquad (4)$$

where: $\mathbf{2\sigma_U}$ is the two sigma uncertainty of M_U (defined above),
$\mathbf{\sigma_T}$ is the counting statistics uncertainty for C_T (defined above),
$\mathbf{\sigma_C}$ is the counting statistics uncertainty for C_C (defined above),
SQRT($(\sigma_T/C_T)^2+(\sigma_C/C_C)^2$) is the counting statistics uncertainty as a fraction of the can count rate,
7/236 is the estimated uncertainty (1 sigma) resulting from the far-field calibration, and
0.0112 is the estimated uncertainty (1 sigma) of the transmission standard.

The Deming assay value is recorded in column nine of Table 2, and the reported value is recorded in column ten. In every case the reported value is just the larger of the two (measured value + 2σ uncertainty). By this conservative technique we were able to give an on-the-spot assay value that included uncertainty and always represented an upper limit of content. The customer requested this conservatism with no reporting uncertainty. One single precise value of HEU content was manifested for each scrap can.

2.2 Deming Interactive Quadratic Curve Measurement

The acquisition of the last six pairs of data in Table 1 gave us the idea to obtain an empirical curve of HEU content that internally contained the sample transmission correction. Each sample acquisition was in a fixed geometry in a constant container. Each sample residue was approximately constant, so that sample self-absorption was directly related to sample content. We therefore reasoned that a single calibration curve ought to be suitable to directly relate measured detection rate to HEU content.

We acquired the data for the initial Deming calibration curve using the WT20xyAic data listed in Table 1. The data are the in-can data of Table 1, where in each acquisition the xy values denote the integer values that identify the standard. These standards are listed and identified in Table 3. In Table 3 we also list the measured cps for each spectrum as well as the known HEU content for each sample. The data of Table 3 are plotted in Figure 6 along with the quadratic best fit to the data. This analysis technique is also described and used in references 3 and 6.

Table 3. Initial Deming Data.

Source	Mass U-235	Gross 185 KeV	Net Area 185 KeV	1σ	Count Time (s)	cps
WT2023Aic	0.69 g inside the std can	1319	932	31	600	1.55
WT2021Aic	4.20 g inside the std can	6157	5538	78	600	9.230
WT2014Aic	1.01 g inside the std can	1738	1319	37	600	2.198
WT2016Aic	39.11 g inside the std can	36582	34370	200	600	57.283
WT2019Aic	15.67 g inside the std can	18952	17359	141	600	28.932
WT2026Aic	98.31 g inside the std can	34600	31708	193	300	105.69
WT2022Aic	9.90 g inside the std can	2500	2477	55	144	17.20

The data in Table 3 and Figure 6 represent the initial acquisition of this Deming interactive fit curve. We reacquired data to obtain an updated curve on four occasions while assaying scrap cans in the time frame from April 2000 to June 2001. We do not present these calibration curves, but note that the data are shown in blocks of acquisitions in Table 2 dated 8 August 2000, 23 August 2000, 31 October 2000, and 4 June 2001.

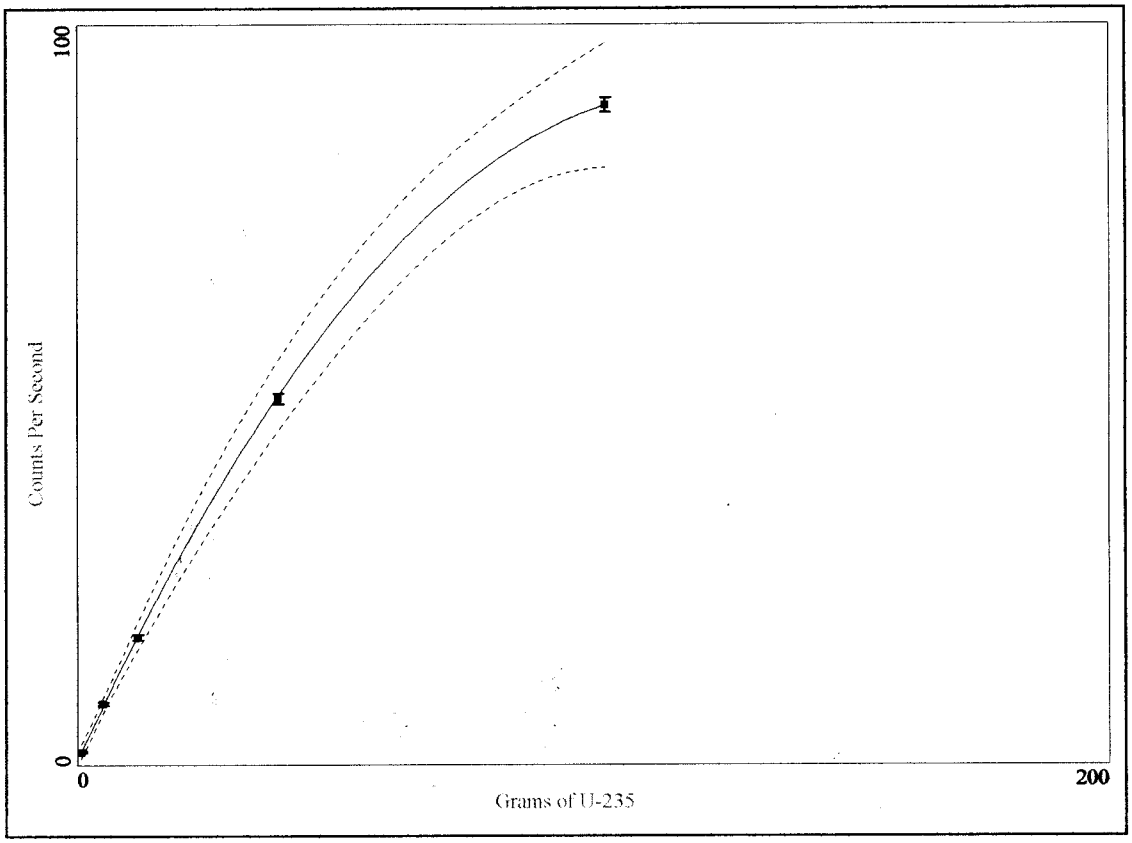

Figure 6. Deming Calibration Curve Used to Obtain ^{235}U Gram Value.

Table 2. Measured sample and transmission data for the 321-M scrap cans assayed for HEU content in this report.

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	t_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
4/25/00	6203	1473	5322±77	2353	339±21	1.48	0.075±0.018	0.1±0.45	0.1
4/25/00	6402	300	1876±49	300	765±29	1.43	1.28±0.130	1.4±0.4	1.2
4/26/00	5506	600	2307±49	600	151±14	1.46	0.129±0.033	0.15±0.45	0.2
4/26/00	6714	300	1120±34	600	109±12	1.47	0.093±0.038	0.11±0.45	0.1
4/26/00	3503	300	1829±44	600	1244±37	1.38	1.00±0.090	1.15±0.38	1.5
4/26/00	6712	300	1130±38	600	82±27	1.46	0.02±0.04	0.1±0.45	0.03
5/3/00	6713	309	1984±47	576	1680±44	1.47	1.51±0.127	1.58±0.24	1.6
5/3/00	8510	300	1838±45	600	1475±41	1.44	1.24±0.108	1.33±0.20	1.5
5/3/00	6203	301	1647±43	600	1486±40	1.60	1.39±0.119	1.33±0.20	1.5
5/3/00	5202	300	2094±48	406	1630±42	1.60	2.26±0.186	2.17±0.33	2.5
5/3/00	5203	304	2507±53	309	1554±41	1.54	2.72±0.225	2.73±0.41	3.1
5/3/00	5204	305	927±33	600	7±8	1.59	0.01±0.042	0.02±0.46	0.03
5/3/00	5205	301	2001±47	600	2017±49	1.52	1.80±0.146	1.82±0.27	2.1
5/31/00	5206	316	2764±55	600	3773±64	1.76	3.88±0.280	3.43±0.51	3.9
5/31/00	5207	305	980±33	600	127±15	1.59	0.12±0.047	0.11±0.02	0.12
5/31/00	5208	541	10202±110	600	9942±107	1.83	10.61±0.711	9.27±1.36	10.6
5/31/00	5209	305	773±30	600	99±12	1.79	0.10±0.055	0.09±0.01	0.1
5/31/00	5210	304	875±32	7200	483±30	1.65	0.04±0.043	0.04±0.01	0.04
5/31/00	5211	300	1364±39	600	1279±37	1.78	1.33±0.119	1.15±0.17	1.3
5/31/00	5212	300	2773±57	314	2233±51	1.89	4.72±0.370	3.88±0.58	4.7
5/31/00	5213	300	2301±51	600	3110±62	1.75	3.18±0.240	2.81±0.42	3.18
5/31/00	5214	300	2705±56	302.96	2291±52	2.29	6.07±0.475	4.13±0.61	6.1
6/1/00	5215	600	2129±50	600	537±25	1.69	0.53±0.066	0.48±0.07	0.5
6/1/00	5216	396	2466±52	600	1682±43	1.49	1.47±0.122	1.51±0.23	1.5
6/1/00	5220	311	1177±37	600	469±24	1.59	0.44±0.063	0.42±0.06	0.5

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	t_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
7/6/00	706canBKG			300	80±18				
7/6/00	ic2021	300	2492±53					4.55±0.68	
7/6/00	stdcanT	300	902±33	300	80±18	1.67	0.16±0.083		
7/6/00	To	300	2210±50						
7/6/00	wt2015	300	5637±81	300	4862±72	1.72	9.77±0.691	10±1	
7/6/00	5305	300	686±29	300	520±29	3.71	2.26±0.310	0.96±0.38	2.3
7/6/00	5306	300	1259±37	300	703±30	2.03	1.67±0.182	1.40±0.11	1.7
7/6/00	5307	300	1346±39	600	1465±42	1.93	1.65±0.147	1.49±0.12	1.7
7/6/00	5308	300	1528±42	300	791±29	1.76	1.63±0.162	1.61±0.13	1.7
7/6/00	5309	300	1198±37	600	1427±41	2.17	1.81±0.162	1.32±0.37	1.9
7/6/00	5310	300	1254±38	600	1899±47	2.74	3.04±0.252	1.76±0.36	3.2
7/12/00	712ToQC	323	2287±54						
7/12/00	stdcanT712	300	892±32						
7/12/00	712canBKG	300			80±22				
7/12/00	5311	300	5625±83	300	4783±79	1.65	9.22±0.659	9.63±1.22	9.9
7/12/00	5312	300	9598±111	300	8734±105	1.63	16.62±1.127	19.19±1.5	20.7
7/12/00	5313	600	17283±145	3600	93093±343	1.61	14.59±0.932	16.86±1.13	18.0
7/12/00	5314	300	11157±124	300	10148±118	1.51	17.86±1.207	22.67±1.75	24.2
7/12/00	WT2021ic			300	2580±54				
7/12/00	WT2019b	100	2904±59	100	2576±58	1.52	13.78±1.072	22.67±1.75	
7/12/00	5314b	100	3649±73	100	3356±69	1.61	18.99±1.435	22.67±1.75	24.2
7/12/00	5315(F Cart)	300	26516±186	300	25883±183	1.90	57.53±3.737	78.74±9.32	88
7/12/00	5316	300	12293±124	300	11523±120	1.72	23.22±1.550	26.17±2.03	28.2
7/12/00	5317	300	4088±71	300	3176±63	1.58	5.88±0.440	6.59±0.53	7.1
7/12/00	712ToQC1	100	710±28						

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	t_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
8/8/00	88canBKG	300	78±21	0.26	0.07				
8/8/00	88ic2021	300	2408±56	7.77	0.2				
8/8/00	88ic2016	178.4	8198±102						
8/8/00	88ic2019	238.8	6293±88						
8/8/00	88ic2015	300	4673±80						
8/8/00	88ic2023	300	429±31						
8/8/00	88ic2026	153	14494±135						
8/8/00	2021canT	300	1026±40						
8/8/00	2021T	300	2243±52	300	1026±40				
8/8/00	5402	300	5734±83	300	4776±77	1.51	8.07±0.575	10.55±2.56	13
8/8/00	5403	300	1522±43	300	441±23	1.42	0.70±0.090	0.93±0.24	1.2
8/8/00	5404	300	11402±126	300	10518±119	1.57	18.51±1.246	24.6±6	31
8/8/00	5407	300	1571±44	600	1328±46	1.55	1.15±0.112	1.41±0.35	1.8
8/8/00	5406	300	1153±38	300	246±19	1.55	0.43±0.079	0.52±0.13	0.6
8/8/00	5404	300	11402±126	300	10335±118	1.43	16.55±1.116	24.75±6.05	
8/8/00	5408	138.2	3673±68	246.7	5657±84	1.41	10.88±0.762	15.5±3.8	19.3
8/8/00	5409	300	5304±81	300	4196±74	1.40	6.60±0.479	9.22±2.25	11.4
8/8/00	5410	159	5335±85	300	9090±112	1.49	15.22±1.036	21±5	23
8/8/00	5405	118.5	5562±87	183.3	8262±105	1.97	29.93±2.045	33±8	36
8/8/00	5401	332.1	2083±50	300	829±33	1.44	1.34±0.138	1.76±0.44	2.2
8/8/00	5315 (Chip Comp)	149.6	5609±84	1800	60808±282	1.40	15.89±1.019	24±6	26
8/8/00	5318	171.4	5265±79	190.9	5309±81	1.58	14.78±1.040	18.72±2.38	21
8/8/00	5319	151.04	5108±82	156.5	4888±79	1.68	17.60±1.253	21.9±2.8	23.6
8/9/00	2021Tqc	300	2151±49						
8/8/00									
8/8/00	2021T	300	2243±52	300	1026±40	1.34	1.54±0.156	1.91±0.36	
8/9/00	5320	300	7950±101	175.4	4189±75	1.67	13.38±0.974	15.43±1.98	18
8/8/00	5218	300	950±34	300	58±21	1.56	0.10±0.083	0.11±0.02	0.2

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
8/23/00	823canBKG	300	88±20						
8/23/00	823QC2021	300	2272±51						
8/23/00	823canT	300	853±32						
8/23/00	8232021To	300	1898±45						
8/23/00	8322014	300	516±26						
	8322016	300	14572±136						
	8322015	149.6	2164±52						
	8322026	55.4	4458±78						
	8322019	300	7196±95						
	8322021a	300	2328±52						
8/23/00	5501a	130.36	6060±89	165.1	7064±99	1.40	21.07±1.46	32.43±4.16	
8/23/00	5502	156.1	7495±98	300	13370±133	1.45	22.73±1.51	34.43±4.77	36.4
8/23/00	5502T1	130.36	6060±89	82.4	3520±67	1.39	20.84±1.54		
8/23/00	5503	85.32	2524±56	75.9	1969±51	1.42	12.88±1.06	16.91±2.07	18
8/23/00	5504	181	1036±37	300	864±34	1.60	1.62±0.12	1.62±0.28	2
8/23/00	5411	105.8	1781±49	150.02	1991±50	1.43	6.66±0.54	7.92±1.21	9.4
8/23/00	5412	300	1774±46	300	1034±38	1.72	2.08±0.20	1.94±0.34	2.4
8/23/00	8235501	159.9	6992±96	300	12235±128	1.57	22.51±1.50	30.27±3.63	34.6
8/28/00	5416	264.2	3935±75	300	3599±71	1.53	6.42±0.48	6.66±0.50	7.2
8/28/00	5417	233.3	4034±76	287.24	3927±75	1.36	6.54±0.48	7.67±0.57	8.2

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	${}^{235}U$ g +2σ	Reported Value
8/28/00	2021ic829			220	1922±51				
8/28/00	2019ic829			228.98	5873±87				
8/28/00	2023ic829			302.22	491±32				
8/28/00	2014ic829			215.26	444±30				
8/28/00	2015ic829			181.68	3069±63				
8/28/00	2016ic829			181.98	8388±105				
Freon™ Cart									
8/28/00	829canT_0	300	2023±52						
7/12/00	5315 (F Cart)	300	26516±186	300	25883±183	1.90	57.53±3.737	78.74±9.32	88
8/28/00	5413(F Cart)	300	11886±128	207.46	7642±103	1.56	20.11±1.38	25.58±1.88	27.5
8/28/00	5414(F Cart)	146.14	6040±90	121.8	4618±80	1.40	18.68±1.35	26.73±2.05	28.8
8/28/00	5415(F Cart)	169.18	5049±80	152.08	4089±76	1.54	14.25±1.05	16.70±1.14	17.8
Cyclone Separator									
8/28/00	Cscan1	300	2712±59	300	1406±47	1.24	2.05±0.19	2.49±0.19	2.7
8/28/00	Cscan2	300	1629±47	300	224±26	1.20	0.31±0.08	0.39±0.03	0.4
8/28/00	Cscan3	300	1658±48	300	204±26	1.18	0.28±0.08	0.35±0.03	0.4

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
6/4/01	321bkg604	6000	1066±69						
6/4/01	Ic2023a	300.48	1077±37	600	870±37	1.65	0.75±0.09	0.55±0.34	
6/4/01	Ic2021			400	3335±62			4.76±0.50	
6/4/01	Ic2014a	300	1483±42	500	1155±39	1.49	1.08±0.10	1.06±0.33	
6/4/01	Ic2016a			200	1051±110			36.93±3.80	
6/4/01	Ic2019a	300	8451±100	300	7943±96	1.85	15.46±1.05	16.71±1.75	
6/4/01	Ic2026a	100	10069±107	100	9852±106	1.54	47.79±3.20	83±12	
A-Lathe									
6/4/01	W0412(Lathe)	300	14563±131	300	13920±129	1.65	24.08±1.59	31.7±3.3	
6/5/01	Tcan	300	822±32						
6/5/01	6205(Lathe)	300	3213±62	300	2499±55	1.56	4.10±0.32	4.76±0.50	5.3
6/4/01	6212(Lathe)	300	9762±107	300	8898±103	1.42	13.28±0.90	18.45±1.98	20.4
6/4/01	6213(Lathe)	300	6534±87	300	5786±86	1.53	9.28±0.65	11.81±1.22	13.0

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
10/31/00	STDcan	500	1711±51						
10/31/00	2022ic	144	2477±55						
10/31/00	2023ic	200.3	347±25						
10/31/00	2021ic	170.28	1414±42						
10/31/00	2026ic	122.86	10963±117						
10/31/00	2016ic	135.26	6698±92						
10/31/00	canbkg1031	1839	292±53						
10/31/00	5101	178.74	1300±43	300	1254±44	1.48	2.169±0.213	2.7±0.4	3.1
10/31/00	5103	300	14915±140	225.4	10668±117	1.68	27.96±1.876	35.3±5.5	41
10/31/00	3503	300	1528±45	300	580±33	1.46	0.99±0.133	1.22±0.19	1.4
10/31/00	5302	300	21904±167	197.12	14138±134	2.29	57.63±3.814	62.53±10.84	73
11/1/00	5105	300	1643±47	300	664±34	1.44	1.12±0.138	1.4±0.2	1.8
11/1/00	5102	174.62	1582±46	300	1827±51	1.51	3.23±0.274	3.9±0.6	4.5
11/1/00	5104	300	16262±148	139.44	7075±98	1.40	24.87±1.721	38.59±5.99	44
11/1/00	5303	300	1415±45	300	605±32	1.58	1.12±0.143	1.28±0.2	1.5
11/1/00	5304	263.3	1715±49	300	1067±39	1.51	1.89±0.185	2.27±0.36	2.6
11/2/00	5201	154.1	440±26	300	56±20	1.59	0.10±0.100	0.12±0.02	0.2
11/2/00	5217	102	1566±44	166.68	2069±53	1.52	6.61±0.540	8.12±1.28	9.4
11/2/00	5219	206.1	1046±37	300	486±32	1.40	0.80±0.121	1.03±0.16	1.2
11/2/00	5421	139.56	3153±66	74.22	1448±44	1.48	10.14±0.891	13.06±2.05	15.1
11/2/00	5418	155.86	487±28	300	50±21	1.51	0.09±0.096	0.11±0.02	0.2

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	M_U = ^{235}U (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
1/10/01	0110BKG	1800	247±29						
1/10/01	5221	300	3676±67	300	2671±51	1.50	4.69±0.56	5.12±0.63	4.7
1/10/01	5106	300	21138±165	300	20318±162	1.66	39.49±2.58		38.8
1/10/01	5507	300	9679±108	300	9001±104	1.83	19.24±1.30	20.7±2.62	18.8
1/10/01	5508	300	5848±85	300	4731±76	1.42	7.88±0.56	9.51±1.21	7.7
1/10/01	5302	300	8298±98	300	6077±96	1.01	7.18±0.51	12.67±1.63	9.8
1/10/01	qc011001	300	2265±53						
1/16/01	11601canBKG	1800	809±40						
1/16/01	11601QC2021	300	2167±50						
1/16/01	5506	300	1229±37	300	70±9	1.43	0.12±0.04	0.14±0.45	0.1
1/16/01	5419	300	1787±46	300	720±30	1.49	1.25±0.13	1.34±0.37	1.2
1/16/01	5107	300	8018±99	300	6726±92	1.35	10.63±0.73	14.29±1.83	11.4
1/16/01	5505	300	1907±47	300	876±33	1.51	1.55±0.16	1.63±0.36	1.5
1/16/01	5420	300	5454±82	300	4591±74	1.65	8.87±0.63	9.20±1.16	8.6
1/16/01	6008	300	1140±36	300	36±7	1.46	0.06±0.04	0.08±0.45	0.1
1/16/01	11601QC2021b	300	2144±50						

Date	Can Number	t_T = CT (sec) 4.41 g U+can	C_T = counts 4.41 g U+can	T_C = CT (sec) Can	C_C = counts Can	Trans Corr	$M_U = {}^{235}U$ (g) Can+2σ	Deming ^{235}U g +2σ	Reported Value
1/17/01	11701canBKG	600	254±22						
1/17/01	11701QC2021	300	2098±52						
1/17/01	6510	300	994±35	300	141±13	1.66	0.27±0.07		
1/17/01	6504	300	1051±37	300	163±15	1.63	0.31±0.07		
1/17/01	6511	300	1019±36	300	73±9	1.58	0.13±0.05		
1/17/01	6505	300	1132±37	300	58±10	1.48	0.10±0.05		
1/17/01	6503	300	1168±36	300	71±10	1.47	0.12±0.05		
1/17/01	6509	300	1158±36	300	84±10	1.48	0.15±0.05		
1/17/01	6506	300	1146±37	300	120±12	1.52	0.21±0.06		
1/17/01	6507	300	1166±36	300	91±11	1.48	0.16±0.05		
1/17/01	6508	300	1180±37	300	72±9	1.46	0.12±0.05		
1/17/01	6512	300	1144±36	300	158±14	1.55	0.29±0.06		
Sum of empty scrap cans 6503-6512							**1.87±0.19**		
1/17/01	11701QC2021b	300	2197±50						

3. DISCUSSION

As shown in Table 2, most of the scrap cans we assayed contained HEU residue in the range of 0.5 to 5 g. Most of the cans were assayed with a count time of 300 sec, for which we obtained a lower limit of content of 0.3 g ^{235}U. Almost all of the transmission correction factors obtained were in the range of 1.4 to 2. From a thorough review of Table 2, the reader can see that about 75% of the time the transmission corrected value and the Deming value agree within 2σ uncertainty. The exceptions are always for the cans that contain bulk quantities of HEU, where we expect the accuracy for both techniques to degrade. For assay results above 20 g, we generally accepted the Deming result. For those below 20 g the two techniques are in 2σ agreement, but we believe the transmission-corrected value provides better accuracy and clearly better precision.

The good agreement between the two techniques of assay lends good support to the reliability of the two assay calculations. But there are several sets of cans that taken together provide especially interesting sets of data and results. We discuss those more thoroughly in this section. The first group is a set of cans assayed by both methods on 4 June 2001. The results for these are listed by that date on page 21 in Table 2. These assays consisted of using the standard can with four selected HEU sources of known content placed sequentially inside the can. The four were then assayed using the Deming interactive curve, and they were assayed using the transmission-correction technique of section 2.1. These are the only four "samples" where we made such a comparison using the known standards available to us.

The four standards assayed inside the standard can are wt2023a, wt2014a, wt2019a, and wt2026a, which have HEU contents of 0.69 g, 1.01 g, 15.78 g, and 98.38 g respectively. We have listed the results for the standards in Table 4 below. Note the assayed values for the first three by both techniques have excellent agreement with the known standards. The assay value for the fourth standard (wt2026a) had poor agreement with the known content because it is a lump sample. Transmission-correction assays by γ-PHA for lump samples are widely accepted by the NDA community to yield results that are biased low. However the results for Table 4 demonstrate the utility of our assay methods in the range of 0.6 g HEU to 16 g HEU.

Table 4. Reported Values for Assay of HEU Standards by Both Techniques.

Source ID	^{235}U Mass (g)	Assay Result (g)	Deming Result (g)
wt2023a	0.69	0.75±0.09	0.55±0.34
wt2014a	1.01	1.08±0.1	1.06±0.5
wt2019a	15.78	15.46±1.05	16.71±1.75
wt2026a	98.38	47.79±3.2	83±12

The next group of scrap cans we discuss is a set of four that were filled with HEU residue obtained in the decontamination of the 321-M A-Lathe.[7] These four cans were assayed on 4 June 2001 and are listed by that date on page 21 in Table 2. We believe the assay results for these four cans represents some of the strongest support for the accuracy of our methods that we present in this report. We refer to scrap cans w0412, 6205, 6212, and 6213 in Table 2.

Scrap can w0412 was filled with HEU residue recovered from the decontamination of the 321-M A-lathe and was placed in 55-gallon drum FD2189 along with job control material from the decontamination work. Drum FD2189 was assayed on the 313-M Q^2 assay instrument in May 2001, and the measured content was 24±14 g HEU. Subsequently drum FD2189 was opened, and scrap can w0412 was removed. The drum was re-sealed and re-assayed with the resulting content of 0.3±0.2g HEU. We conclude the 24±14 g from the Q^2 assay came almost entirely from can w0412.[7]

Scrap can w0412 was then assayed on the far field scrap can assay station of this report. As seen in Table 2, the transmission corrected result was 24.08±1.59 g, and the Deming result was 31.7±3.3 g. Both results are in good agreement with the Q^2 result and are in fairly good mutual agreement. The measured transmission correction factor was 1.65.

Because can w0412 contained more than 20 g of HEU, we repacked its contents into three cans with the intention to assay the three cans each within the range of 0 to 15 g HEU. The contents were packed into cans 6205, 6212, and 6213, and each was assayed on the far field station. As seen in Table 2, the results provide extremely good support for the original measurement of can w0412 and are in very good mutual agreement.

Finally to emphasize this point even more, each of the cans 6205, 6212, and 6213 were placed into separate 55-gallon drums and were assayed again. The results are reported in Table 5 of reference 7 and are 3±2 g, 10±6 g, and 7±4 g respectively. Once again the results were in excellent agreement with the transmission-corrected assays of the far field assay station.

The third group of scrap cans we discuss is a set of four cans filled with recovered residue from the decontamination of the 321-M Freon™ cart.[8] All of the recovered residue from this decontamination was placed originally in scrap can 5315, which was assayed on 12 July 2000 as shown on pages 17 and 20 in Table 2. The transmission-corrected result for can 5315 was 57.53±3.74 g with a correction factor of 1.90, and the Deming interactive fit result was 78.74±9.32 g. Since both of these values were out of our preferred range, the recovered residue was removed from 5315 and re-packed into three separate cans just as above for the A-lathe cans.

These three repacked cans were assayed as cans 5413, 5414, and 5415 on 28 August 2000 and are listed on that date on page 20 in Table 2. The measured values by transmission correction were 20±1 g, 19±1 g, and 14±1 g; while the Deming values were 26±2 g, 27±2 g, and 17±1 g. Note that scrap can 5315 was subsequently filled with material from the chip compactor and re-assayed on 8 August 2000.

All six of the measured values for scrap cans 5413 – 5415 are at the marginal limit of our region of accuracy, but the two values for each can agree within 30%, and the sums agree within 24%. Consistent with our policy to accept the Deming results for cans that assayed above 20 g, we believe the Deming values for these four cans to represent the more accurate results, and those are the values we reported in reference 8. These values demonstrate that approximately 70 g of HEU residue was recovered from the decontamination of the 321-M Freon™ cart. This value is consistent with the holdup assays reported in reference 8 before and after decontamination of the cart.

A fourth set of three cans we discuss contains the scrap cans from the cyclone separator. These three cans contain residual HEU recovered from the decontamination of the cyclone separator and are labeled CScan1, Cscan2, and Cscan3. They were assayed on 28 August and are listed by that date on page 20 in Table 2. These three cans have a geometry and container thickness different from all of the other scrap cans and from the standard can. A photograph of one of these cans is shown in Figure 7. Note the transmission correction factors for these three cans ranged from 1.18 to 1.24, which is significantly lower than the correction factors for the larger scrap cans.

The first observation for these three cyclone separator cans is that the measured transmission correction factors are in a very small range. That is, they have extremely good precision. We believe this is a good endorsement of the assay technique. A second observation is that the measured values by the two techniques are in good mutual support. The transmission corrected results for the three cans are 2.05±0.19, 0.31±0.08, and 0.28±0.08; and the Deming results are 2.49±0.19, 0.39±0.03, and 0.35±0.03. Since the Deming technique was calibrated with the standard can, it is not completely applicable to these three cans. However the two techniques *still* yielded values in very good agreement. These three cans lend especially strong support to the transmission corrected assay technique.

Figure 7. A photograph of a cyclone separator can.

4. RESULTS

Table 5 contains a list of all of the scrap cans assayed and grouped by component whence the HEU residue came.[9] In particular in Table 5 we have three groups of cans that came from the Freon™ Cart, the A-Lathe, and the Casting Room. For each of these components we have a single can that contained excessive amounts of HEU that we requested the 321-M operators to repack into multiple cans. In every case the sum of the content in the repacked cans agrees well with the assayed content in the original can – even when the content of the original can was outside of our accepted calibration range.

The A-lathe and Freon cart scrap cans have already been discussed in this report. The casting room cans are numbers 5302, 5104, 5507, and 5107 as identified in Tables 2 and 5. The assayed sum in the repacked cans 5104, 5507, and 5107 matches exactly the reported content in the original casting room can 5302. Can number 5104 is identified in reference 9. It was sent to Solid Waste in B-25 container #1883, and we are pretty sure it is a repacked casting room can.

Table 5. A list the reported values of scrap cans grouped by component.

Assay Date	Can Number	Major Component	Reported ^{235}U Content
11/1/00	5104	Not known	44
10/31/00	5302	Casting Room	73
1/10/01	5507	Casting Room	18.8
1/16/01	5506	Casting Room	0.1
1/16/01	5107	Casting Room	10.2
4/25/00	6402	Machine Room	1.2
1/10/01	5221	Machine Room	4.2
1/10/01	5508	Machine Room	7.7
1/16/01	5419	Machine Room	1.2
1/16/01	5505	Machine Room	1.5
1/16/01	5420	Machine Room	8.6
1/16/01	6008	Machine Room	0.1
8/8/00	5401	Machine Room Work Table	2.2
11/2/00	5219	Machine Room Floor	1.2
8/8/00	5405	Machine Room Floor	36.0
11/2/00	5217	Machine Room Cart	9.4
11/2/00	5201	Decon Machine Vac	0.2
11/2/00	5421	B Lathe	15.1
11/2/00	5418	B Lathe	0.2
8/23/00	5504	B Lathe	2.0
8/8/00	5218	B Lathe	0.2
8/23/00	5412	A/C Furnace	2.4
8/23/00	5411	C Furnace	9.4
10/31/00	3503	C Casting Furnace	1.4
1/10/01	5106	C Furnace	38.8
11/1/00	5105	B Furnace	1.8
10/31/00	5103	B Furnace Floor Sweepings	41
11/1/00	5102	B Cyclone Separator	6.0
11/1/00	5304	Can Vault Machine	2.6
10/31/00	5101	Floor Sweepings	3.1
10/31/00	5303	Casting Room Sweepings	1.5

Date	Can Number	Major Component	Reported ^{235}U Content
8/8/00	5315	Chip Compactor	26.0
8/8/00	5318	Chip Compactor	21.0
8/8/00	5319	Chip Compactor	23.0
8/9/00	5320	Chip Compactor	18.0
7/6/00	5305	Chip Compactor	2.3
7/6/00	5306	Chip Compactor	1.7
7/6/00	5307	Chip Compactor	1.7
7/6/00	5308	Chip Compactor	1.7
7/6/00	5309	Chip Compactor	1.9
7/6/00	5310	Chip Compactor	3.2
7/12/00	5314	Chip Compactor	24.2
7/12/00	5316	Chip Compactor	28.2
7/12/00	5317	Chip Compactor	7.1
7/12/00	5311	Chip Compactor	9.9
7/12/00	5312	Chip Compactor	20.7
8/28/00	5413(F Cart)	Freon™ Cart	27.5
8/28/00	5414(F Cart)	Freon™ Cart	28.8
8/28/00	5415(F Cart)	Freon™ Cart	17.8
8/28/00	5416	NC Lathe	7.2
8/28/00	5417	NC Lathe	8.2
8/23/00	5501	NC Lathe	34.6
8/23/00	5503	NC Lathe	18
8/23/00	5502	NC Lathe	36.4
8/8/00	5406	NC Lathe	0.6
8/8/00	5409	NC Lathe	11.4
8/8/00	5410	NC Lathe	23.0
8/8/00	5404	Log Saw	31.0
8/8/00	5407	Log Saw	1.8
8/8/00	5402	Log Saw	13.0
8/8/00	5403	Log Saw	1.2
6/5/01	6205(Lathe)	A-Lathe	5.3
6/4/01	6212(Lathe)	A-Lathe	20.4
6/4/01	6213(Lathe)	A-Lathe	13.0
8/8/00	5408	Sieving Hood	19.3

5. CONCLUSION

Using a portable HPGe detector with an EG&G DART® electronics package and an Ortec Gamma-Vision acquisition system and three personal computers, we have assembled a scrap can assay station in Building 324-M to determine HEU content in fixed geometry scrap cans. The scrap cans have been filled during the FDD two-year campaign to deactivate and decommission (D&D) Building 321-M. During the D&D campaign, HEU residue recovered from process equipment and from the building floor and exhaust system was packaged into two-gallon scrap cans for subsequent burial as low level waste. Each scrap can was assayed for both criticality safety purposes and MC&A purposes.

The assay station is a far field transmission-corrected γ-PHA acquisition system that has been efficiency calibrated in two configurations. It was assembled and operated in a resourceful and efficient manner that allowed us to deliver on-the-spot assay results from both acquisition configurations. Using this assay station we have determined the HEU content of 108 scrap cans that had contents ranging from <0.1 g up to 88 g. One of the assay configurations is the traditional point source transmission correction method. The other configuration is one we developed on-site. For this configuration we experimentally obtained a constant-geometry, constant-transmission calibration curve from eight standards assayed from a distance of 48". These eight standards yielded an empirical least-squares-curve that we fit with a three-parameter quadratic function using Deming software.

The assay station has proven especially valuable to confirm the holdup assays performed on the 321-M Freon™ Cart and on the 321-M A-Lathe. Decontamination activities of both of these items of process equipment yielded amounts of HEU residue near 80 g. In both instances by assaying all of the residue in a single can and then splitting the residue into three separate cans and re-assaying, we were able to confirm the utility of the assay station over the entire mass range. This technique of splitting was used to confirm the assays in the upper end of our mass range and served as excellent support of the transmission correction measurements made. The technique of splitting was also very useful for confirmation of assays of scrap cans filled with residue from the casting room.

6. REFERENCES

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