DEVELOPMENT AND CHARACTERIZATION OF A CHAMBER GRAM ESTIMATOR

by

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July 1995

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Gram Estimator
Line Generated waste
Nondestructive Assay
Plastic Scintillator

ROCKY FLATS ENVIRONMENTAL TECHNOLOGY SITE
TECHNOLOGY DEVELOPMENT
P. O. BOX 464
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TECHNICAL REVIEWS

This report has been reviewed for content by a qualified subject matter expert (SME) and the conclusions and recommendations have been read and understood by the undersigned.

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ABSTRACT

The Chamber Gram Estimator (CGE), Model SAM-1B is a small article gamma monitor originally designed to screen items for radioactive contamination that may be placed into its chamber. Four plastic scintillator detectors detect photons emitted from bulk quantities of fissile materials in line generated waste and provide a fissile mass content estimate of waste packages. With the present CGE, a few grams of ^{239}Pu could be distinguished above background in light matrix samples. It is controlled by a personal computer that uses a menu driven operating program. The program is designed to reduce the potential for operator error while obtaining a fissile material gram estimate of a line generated waste package prior to placement in a drum.

This report describes the work performed to characterize the counting chamber for fissile material estimation. The operating features of the CGE, the results and conclusions drawn from the experiments, and the future work recommended for the next generation CGEs are also described.

The CGE provides advantages over hand-held Ludlum 12-12 survey-type instruments presently in use. The CGE is easy to operate, does not require manual manipulation to measure the total surface of the sample, and provides a display free of rate meter response-time effects or the need for visual averaging. Moreover, the response variations due to sample geometry are significantly less than with a hand-held arrangement, particularly, for the low density matrix samples appropriate for estimation. The waste packages are placed inside the instrument's shielded chamber which effectively eliminates gamma exposure to operators from waste packages.

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TABLE OF CONTENTS

Technical Review.....	ii
Abstract.....	iii
Acknowledgements.....	vi
Table of Contents.....	v
 1. INTRODUCTION.....	 1
2. DESCRIPTION OF CHAMBER GRAM ESTIMATOR.....	2
3. THEORETICAL CONSIDERATIONS.....	4
4. EXPERIMENTS PERFORMED ON THE CGE.....	4
4.1 Preliminary Electronics and Internal Shield Settings...	4
4.2 Calibration of the CGE.....	5
4.3 CGE Source Position Characterization Study.....	23
4.4 Other Characterization Studies Performed on CGE.....	39
5. OPERATION OF THE CGE.....	39
6. RESULTS AND DISCUSSIONS.....	41
7. CONCLUSIONS AND RECOMMENDATIONS.....	42
8. FUTURE WORK.....	42
9. REFERENCE.....	43
Appendix A - Photographs of the CGE Setup.....	44
A-1. Chamber Gram Estimator Setup.....	45
A-2. Inside of Empty CGE.....	46
A-3. Bagcut Positioned Inside CGE.....	47
Appendix B - Fixed Counts and Fixed Real-Time Pulse Height Spectra.....	48
B-1(a-e). Preset Count Pulse Height Spectra.....	49
B-2(a-e). Fixed Real-Time Pulse Height Spectra....	54

DEVELOPMENT AND CHARACTERIZATION OF A CHAMBER GRAM ESTIMATOR

1. INTRODUCTION

The amount of fissile material in low level line generated waste packages can be estimated with gamma radiation measuring instrumentation that measures gamma rays coming out from the surface of such packages. A Ludlum 12-12 Portable Gram Estimator (PGE) is currently used to estimate plutonium (Pu) mass in such packages which have no defined shape. The PGE is calibrated using a 5 gram tank surveillance standard that has the shape of a disk within a 5 inch diameter plate. A study, "Investigation of Portable Gram Estimators" (January 1992, unpublished), pointed out that the PGE is not conservative when estimating packages containing fissile material in a dense material category or Item Description Code (IDC). The study also indicated that for packages with light matrix IDCs, the estimations of plutonium could be in error by as much as a factor of 3 due to geometry effects alone. The PGE is essentially a rate meter coupled to a Sodium Iodide (NaI) detector. Due to the limited discrimination capabilities of the rate-meter electronics, a 1/16 inch thick cadmium shield is required for the NaI detector to limit the response from americium (^{241}Am) which is present in weapons grade plutonium. It cannot compensate for the waste package matrix absorption or for the errors associated with the geometry of the package. Due to a single adjustment for the PGE rate meter, the instrument display cannot provide direct readings specific for different IDCs, and conversion factors must be applied to the results. Moreover, the risk of exposure to the operator is increased due to the need to hold and rotate the package in front of the operator while obtaining the estimate using the PGE.

Developmental work on the Chamber Gram Estimator (CGE) was initiated as a long term solution to the problems associated with the Ludlum 12-12 Portable Gram Estimator¹. In comparison to the cost of using a Segmented Can Counter, which corrects for density variations in a package, the CGE is an inexpensive and quick method to use in estimating low level line generated waste packages prior to placing them into waste drums. Fissile material estimators must be appropriately characterized for the various waste matrices to avoid violating established criticality safety limits.

This report describes construction of the CGE and the experiments performed to properly set the electronics and to select the internal chamber shield thickness. Calibration of the instrument using different standards and the source position dependence within the chamber were studied. The operation of the CGE, results and conclusions drawn from the experiments, and the future work recommended for the next generation CGEs are also described.

After performing initial electronic adjustments and mapping the chamber using radioactive sources in Building 881, the CGE was shipped to Building 771, Room 114.

2. DESCRIPTION OF CHAMBER GRAM ESTIMATOR

The Chamber Gram Estimator, Model SAM-1B is a prototype small article monitor built by TSA Systems, Longmont, Colorado. It was originally designed to screen items for radioactive contamination that may be placed into its chamber.

The CGE resembles a small refrigerator and has a sample chamber box measuring 15"H x 15"W x 18"D (see Photograph in Appendix A). The outside dimensions of the box are 29"H x 26"W x 26"D, and it weighs about 900 pounds. It has 3/8" thick lead shielding on all six sides of the sample chamber. There are four plastic scintillation detectors measuring 10"W x 15"L x 1.5" thick positioned at the top, bottom, left and right sides of the chamber. The detectors are coupled to four photomultiplier tubes via light pipes. There is a common high voltage bias supply for all four detectors. Figure 1 illustrates a simplified diagram of how counts from two channels are sent to the counter. Signals are conditioned by four individual amplifiers that may be adjusted separately. The amplifier signals are discriminated using two separate single channel analyzers (SCA) each combining the response of two detectors. The two SCAs have common threshold and window controls. There are two counting channels, one for each SCA and its associated detector pair.

The CGE was designed to automatically perform a self-test and acquire a new background count rate each time it is powered up. Initial operation involved the use of a hand held terminal. This protocol has been changed through the use of an adjacent personal computer and a menu driven operating program. The program was designed to significantly reduce the potential for operator error while obtaining a Pu gram estimate of a waste package. An internal shield, 1/8" thick copper sheet, has been installed inside the CGE chamber to minimize ²⁴¹Am response.

FIGURE 1: Block Diagram - CGE Electronics

3. THEORETICAL CONSIDERATIONS FOR THE CGE

Passive techniques used to estimate Pu mass in small packages from the gamma-ray flux emitted, is a function of the flux emitted and the fraction of gamma rays detected. The flux emitted depends on (1) the Pu content of the package, (2) self-absorption in the Pu, i.e., the density and dispersion of the Pu within the waste package matrix, (3) gamma-ray absorption in the waste material matrix in the package, (4) other gamma ray emitting radioisotopes, such as ^{241}Am , in the waste package, and (5) background radiation. Detection of the gamma ray flux is a function of the detector geometry, detector efficiency, energy spectral response, the internal shield to limit the response to low energy photons (due to ^{241}Am), the external shield to limit the background contribution, and dead time of the detector.

Variables associated with the gamma flux emitted were investigated using several sample configurations. The response of the instrument was measured as a function of fissile material mass using small cylindrical standards containing PuO_2 . The measurements provided a conservative indication of linearity, combining the effects of self-absorption, and to a degree, dead time of the instrument. Variables associated with the photon flux detected were investigated using pulse height analysis and relative efficiency measurements.

4. EXPERIMENTS PERFORMED ON THE CGE

4.1 PRELIMINARY ELECTRONIC AND INTERNAL SHIELD SETTINGS

Measurements were performed to study the independent response of all four detectors, and these measurements were used to verify optimum operating parameters (e.g., high voltage, amplifier gains, discriminator settings, region-of-interest).

Pulse height spectra for a preset 400,000 integrated counts for two 5 and 50 milligram ^{241}Am standards, two 5 and 50 gram standards containing PuO_2 , and the background were obtained. These spectra are given in Appendix B and were used to compare electronic efficiencies in determining the optimum region-of-interest (ROI) in combination with amplifier gains and discriminator settings. The data showed a very limited dependence of electronics alignment (ROI) on efficiency for the ^{239}Pu and interferences from the ^{241}Am and background.

Pulse height spectra for a 180 seconds fixed real-time were obtained for the background and four ^{241}Am and PuO_2 standards (5 and 50 gram each) and are also given in Appendix B. The data provided a relative count rate comparisons between sample quantities (^{239}Pu) and interference from the background and ^{241}Am . Measurements at different discriminator settings showed that the factory setting was the optimum setting to minimize the response due to ^{241}Am .

The data demonstrated adequacy of shields, both external and internal, to allow a few grams of ^{239}Pu to be distinguished above background in light matrix materials and to limit response contribution from ^{241}Am at concentrations of 1,000 parts per million (ppm), respectively, in the chosen operating region.

4.2 CALIBRATION OF THE CGE

4.2.1 Using Triple Encapsulated Standard Sources

The instrument responses were measured as a function of plutonium mass using small cylindrical standards containing PuO_2 compressed within a relatively small volume. These triple encapsulated PuO_2 standards (TES) ranged from 5-50 grams of PuO_2 (total encapsulation aluminum tube thickness of 3/16"). These TESs were placed and counted for 100 seconds inside a tube (1/16" thick aluminum) fitted at the central axis of the clamshells (Figure 2). Calculations indicated that, for proton energies from 60 keV to 150 keV, a transmission of only about 65 to 80 percent takes places due to absorption by the aluminum tubes. Self-absorption by the PuO_2 source material in the TESs was not calculated, however, it was higher for sources with a larger amount of source material.

These clamshells were filled with different IDC matrix materials which corresponded to expected waste characteristics and did not contain PuO_2 within the matrix. For the TES sources, counts were performed for 100 seconds each and net count rates were obtained, after subtracting the background count rate (about 4200 cps). Calibration data were plotted as shown in Figures 3 through 15.

4.2.2 Using Clamshell Homogeneous Standard Sources

Six clamshell homogeneous standards (CHS) were fabricated for IDCs 331, 336, 337, 339, 431, and 480 with approximately 50 grams of PuO_2 distributed homogeneously throughout each clamshell matrix. Details of these standards are given in Table 1. Measurements

FIGURE 2

CLAMSHELL MAPPING POSITIONS

FIGURE 3: Calibration Curve for IDC #290 (Filter Sludge)

FIGURE 4: Calibration Curve for IDC #331 (Ful-flo Filters)

FIGURE 5: Calibration Curve for IDC #332 (Oily Sludge)

FIGURE 6: Calibration Curve for IDC #336 (Wet Combustibles)

FIGURE 7a: Calibration Curve for IDC #337 (Plastics)

FIGURE 7b: Calibration Curve for IDC #337 (Polythelene)

FIGURE 7c: Calibration Curve for IDC #337 (Teflon + PVC)

FIGURE 8: Calibration Curve for IDC #338 (Filter Media)

FIGURE 9: Calibration Curve for IDC #339 (Lead Drybox Gloves)

FIGURE 10: Calibration Curve for IDC #375 (Oil Dri)

FIGURE 11: Calibration Curve for IDC #431 (Resin)

FIGURE 12: Calibration Curve for IDC #438 (Insulation)

FIGURE 13: Calibration Curve for IDC #440 (Glass)

FIGURE 14: Calibration Curve for IDC #444 (Ground/Lead Glass)

FIGURE 15: Calibration Curve for IDC #480 (Light Metals)

of these standards were compared with the similar matrix TES calibrations where TES standards were placed at the center in central tube of the clamshells with no PuO_2 in the matrix. Measured count rates for the CHS standards are shown on the corresponding TES calibration graphs.

TABLE 1: CLAMSHELL HOMOGENEOUS STANDARDS

<u>IDC No.</u>	<u>Matrix Description</u>	<u>PuO_2 (g)</u>	<u>From TES Calibration Curve (g)</u>
331	Ful-flo Filters	49.99 ± 0.23	117.0
336	Wet Combustibles	49.99 ± 0.23	123.9
337	Poly, Teflon, PVC	49.61 ± 0.23	150.2
339	Lead Gloves	49.62 ± 0.23	184.1
431	Resin	49.99 ± 0.23	96.3
480	Light Metals	49.89 ± 0.23	140.5

Data for eight available Can Counter Standards (CCS), four each for IDC 331 and 338, are given in Table 2. These CCSs, which had very high ^{241}Am content, were counted in the CGE. Their fissile material mass content estimations, using TES calibration curves for similar matrices, are also given in Table 2.

TABLE 2: CAN COUNTER STANDARDS

<u>IDC No.</u>	<u>Matrix Description</u>	<u>PuO₂ (g)</u>	<u>From TES Calibration Curve (g)</u>
331	Ful-flo Filters	0.00	0.0
		19.9 ± 0.6	51.5
		65.7 ± 0.4	155.1
		138.3 ± 0.4	251.3
338	Filter Media	10.6 ± 0.3	28.7
		15.3 ± 0.4	46.9
		27.4 ± 0.8	77.7
		46.2 ± 0.5	139.2

4.3 CGE SOURCE POSITION CHARACTERIZATION STUDY

A 50 gram TES was positioned at 27 positions around various clamshells filled with different IDC matrix materials. Position numbers were as indicated in Figure 2. Counting was performed for 10 seconds and net count rates were determined in each case. Plots in Figures 16-28 show observed detector responses as a function of the source position around different IDC clamshells.

Data from these plots were used to estimate measurement uncertainties due to source position inside the package or source geometry effect. In a clamshell, for a particular matrix, the ratios between the highest count rate position divided by the center position (Position One) count rate and the center position count rate divided by the lowest count rate position were determined, and a geometry factor or 'G-Factor' for the IDC was calculated as the larger of the two ratios. This factor is used as a multiplier to the Pu mass estimated from the TES calibration curves for different IDCs.

FIGURE 16: Variation of Response with Source Position for IDC #290

FIGURE 17: Variation of Response with Source Position for IDC #331

FIGURE 18: Variation of Response with Source Position for IDC #332

FIGURE 19: Variation of Response with Source Position for IDC #336

FIGURE 20a: Variation of Response with Source Position for IDC #337

FIGURE 20b: Variation of Response with Source Position for IDC #337

FIGURE 20c: Variation of Response with Source Position for IDC #337

FIGURE 21: Variation of Response with Source Position for IDC #338

FIGURE 22: Variation of Response with Source Position for IDC #339

FIGURE 23: Variation of Response with Source Position for IDC #375

FIGURE 24: Variation of Response with Source Position IDC #431

FIGURE 25: Variation of Response with Source Position for IDC #438

FIGURE 26: Variation of Response with Source Position for IDC #440

FIGURE 27: Variation of Response with Source Position for IDC #444

FIGURE 28: Variation of Response with Source Position for IDC #480

4.4 OTHER CHARACTERIZATION STUDIES PERFORMED ON CGE

Matrix absorption effects were characterized with the TES and CHS standards within various matrices constructed from synthetic waste forms. Encapsulated ^{241}Am standards were also used to quantify the response contributions from the PuO_2 standards anticipated with actual waste packages as discussed in Section 4.1.

Background measurements as well as measurements using empty clamshells (no source but filled with different matrices) were also performed. These measurements indicate the lower detection limits that can be expected for a proposed operating location.

5. OPERATION OF THE CGE

A procedure "Chamber Gram Estimator Building 771," 4-B28-CGE-001, has been issued for operation of the CGE. When an operator places a waste package inside the sample chamber and follows the procedure for utilizing the measurement program, the technique dependence that was of concern with the PGE is eliminated and the risk of exposure to the operator is significantly reduced.

After the operator powers up the computer and the CGE, a batch file automatically displays a Main Menu and the operator is then directed to follow the prompts as outlined in the operating procedure. Associated with the operating program is a data file that contains calibration data for each IDC accounting for matrix absorption, a factor accounting for the Pu distribution in the matrix (geometry factor), and a time of estimation for each IDC (Table 3). These parameter data values were determined using TES sources for different matrices based on the expected waste characteristics. Within the operating program, there is an option for the operator to print out the results of the estimate that could be attached to the Waste/Residue Traveler form for the waste drum that is being loaded.

TABLE 3: CALIBRATION DATA FILE

<u>IDC No.</u>	<u>a ($\times 10^{-9}$)</u>	<u>b ($\times 10^{-4}$)</u>	<u>c</u>	<u>Geometry Factor</u>	<u>Count Time (seconds)</u>
290	1.16	5.26	-2.61	1.63	100
330	0.959	2.97	-1.63	1.27	50
331	0.918	2.88	-1.60	1.25	100
332	0.944	5.53	-2.02	1.75	100
335	0.959	2.97	-1.63	1.27	50
336	1.00	2.86	-1.37	1.30	50
337	1.07	4.14	-1.96	1.48	50
338	0.959	2.97	-1.63	1.27	50
339	2.20	7.57	-2.53	2.30	100
341	2.20	7.57	-2.53	2.30	100
342	0.959	2.97	-1.63	1.27	50
375	1.31	3.19	-1.29	1.30	50
376	0.959	2.97	-1.63	1.27	50
431	1.24	3.42	-1.23	1.41	50
438	0.885	3.28	-1.83	1.25	50
440	0.948	3.01	-1.62	1.25	50
444	2.60	1.28	-2.76	3.77	100
480	2.79	5.08	-2.09	2.00	100

6. RESULTS AND DISCUSSIONS

Due to the limited gamma energy resolution of plastic scintillation detectors, electronic discrimination provides no significant optimization of the ^{239}Pu to ^{241}Am response ratio, and very precise electronic adjustments were not necessary. An internal 1/8" Cu shield thickness, in conjunction with threshold settings, was found to be sufficient to prevent significant ^{241}Am response due to the concentrations inherent in the calibration standards used. Also, the external 3/8" lead shield thicknesses was adequate to reduce the background response for all practical purposes even in the ^{241}Am process environment in Building 771.

With the present CGE, a few grams of ^{239}Pu could be distinguished above background in light IDC matrix samples. The CGE has a limited response contribution from ^{241}Am at concentrations of 1,000 ppm at the chosen operating region.

It is evident from the data that the TES calibration curves in Figures 3-15, when used for unknown samples give very conservative estimates of the fissile mass contents. The estimates are about a factor of 2 to 4 overestimation of fissile mass for the homogeneously distributed fissile matrices (CHS). This is due to the construction differences of the TES and CHS sources. In the case of TES standards, absorption by the 1/4" aluminum tube thickness (total source and mounting tube thicknesses) and PuO_2 self-absorption due to compactness of the fissile material, the photon flux reaching the detectors is greatly reduced. In the CHS sources, on the other hand, the material is thinly distributed and there is very little, if any, self-absorption; hence the production of a higher count rate for the same amount of fissile material.

In actual practice, a line generated waste package may not have a homogeneous distribution or a point-source form of the fissile material. Moreover, it is required that the CGE should not underestimate the fissile contents of the package to avoid violating criticality safety limits. It is therefore acceptable to overestimate and not to underestimate the fissile material contents. Although, it is helpful to limit overestimation tolerances, errors such as material matrix and self-absorption are unavoidable with instruments like the CGE.

Basic limitations of passive gamma measurement instruments for fissile material mass determination apply to hand-held instruments as well as counting chamber estimators. Their responses are highly dependent on the density of the fissile fraction of the waste form. Appropriate choice of calibration standard matrices is important to avoid both underestimation and over conservatism. Matrix effects

can be determined empirically, however, estimation uncertainties are large, particularly for higher density IDCs.

7. CONCLUSIONS AND RECOMMENDATIONS

The CGE provides the advantages over the Ludlum 12-12 PGE presently in use. The CGE is easy to operate, does not require manipulation/rotation of the sample during measurement, and provides a discrete display value free of rate meter response-time effects or the need for visual averaging. The chamber shielding effectively eliminates gamma exposure to operators from waste packages during the measurement process. Response variations due to sample geometry are significantly less than with hand-held instruments, particularly, for the low density matrix samples appropriate for estimation.

Fissile material estimation techniques need to be viewed as a supplement to a Non-destructive Assay (NDA) program, and are useful when the rate of waste package generation exceeds the throughput of accountability measurements through NDA systems. In terms of priority, fissile packaging operations should be reviewed for the adequacy of available assay systems. If assay systems are available and a fissile packaging program is expected to generate samples more rapidly than can be conveniently certified, it may be reasonable to consider continuing the practice of estimation. However, estimation results should be routinely compared with assay values to insure the validity of tolerances predicted for various waste forms. NDA specialists should oversee estimation programs to ensure at least subjective equivalency between waste and calibration matrices. In cases where estimation is appropriate and samples typically exceed a few inches in thickness or contribute significant gamma exposure, a counting chamber design should be pursued.

8. FUTURE WORK

If estimation techniques are appropriate for future operation, e.g., liquid stabilization and thermal stabilization, and additional units become desirable, the next generation of counting chambers should be investigated and implemented. Chambers now are available with thicker external shielding, larger detectors completely enclosing the sample volume (one detector in all six walls giving close to true 4π geometry) and reduced chamber

dimensions. The increased detector surface area will improve sample response and geometry effects (G-Factors), and the additional shielding and reduced sample volume will reduce background. These parameter improvements will optimize sensitivity of measurement.

Additional efforts to correlate existing standards to waste matrices, to fabricate additional standards more representative of waste forms, and to better define the tolerances associated with waste variables remain a priority for estimation programs.

9. REFERENCE

1. Dulco, G. B. and Balmer, D. K., "Investigation of Portable Gram Estimators," Rocky Flats Plant, January 1992, Unpublished.

APPENDIX A

PHOTOGRAPHS OF THE CGE SETUP

FIGURE A-1: Chamber Gram Estimator Setup.

FIGURE A-2: Inside of Empty CGE.

FIGURE A-3: Bagcut Positioned Inside CGE.

APPENDIX B

PRESET COUNT AND FIXED REAL-TIME PULSE HEIGHT SPECTRA

FIGURE B-1a: Preset 400,000 Count Pulse Height Spectra - Background.

FIGURE B-1b: Preset 400,000 Count Pulse Height Spectra -
5 Gram PuO_2 Standard Source.

FIGURE B-1c: Preset 400,000 Count Pulse Height Spectra -
50 Gram PuO_2 Standard Source.

FIGURE B-2d: Fixed Real-Time Pulse Height Spectra -
5 Milligram ²⁴¹Am Standard Source.

FIGURE B-1e: Preset 400,000 Count Pulse Height Spectra -
50 Milligram ^{241}Am Standard Source.

FIGURE B-2a: Fixed Real-Time Pulse Height Spectra - Background.

FIGURE B-2b: Fixed Real-Time Pulse Height Spectra -
5 Gram PuO_2 Standard Source.

FIGURE B-2c: Fixed Real-Time Pulse Height Spectra -
50 Gram PuO_2 Standard Source.

FIGURE B-1d: Preset 400,000 Count Pulse Height Spectra -
5 Milligram ^{241}Am Standard Source.

FIGURE B-2e: Fixed Real-Time Pulse Height Spectra -
50 Milligram ^{241}Am Standard Source.