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EVALUATION OF NONDESTRUCTIVE ASSAY SYSTEM
PERFORMANCE ON Z-9 CRIB SOIL MEASUREMENTS
PHASE I

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PHASE I

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1.0 INTRODUCTION

Plutonium-contaminated soil being removed from the Atlantic Richfield Hanford Company (ARHCO) 216-Z-9 Crib Mining Facility is being packaged in nine-liter quantities, in double metal canisters, for eventual storage or recovery. Plutonium content per canister package is measured nondestructively with a Sodium Iodide (NaI) gamma detector and associated electronics system known as the Soil Assay Monitor System (SAMS). The SAMS is installed at the Z-9 Facility and the canisters are measured immediately after being removed from the glovebox and prior to further containment in fifty-five gallon metal barrels.

An analytical verification plan was established as part of the Facility start-up program to provide for remeasurement of the first fifty canisters by an independent nondestructive assay (NDA) instrument. The Segmented Gamma Scan Assay System (SGSAS), located in the 234-5Z building, is considered to be the most advanced gamma measurement system available for the routine determination of plutonium-contaminated waste and scrap materials in their shipping and storage containers. The accuracy and precision of the technique is well-documented (reference 13).

The analytical plan is divided into three phases, as follows:

- I. The first sixteen canisters will be checked individually on both systems. Following the accumulation of 16 pieces of data, the performance of both systems will be evaluated.

The Manager of 234-5 Laboratory will issue a report summarizing the evaluation.

- II. Canisters 16-50 will be measured individually on both systems.

A second summary report will be issued after evaluation of this data.

- III. After canister 50, every 10th canister will be measured on both systems.

All canisters containing 11.5 grams or more plutonium will be measured on both systems regardless of the sampling plan.

Phase I has been completed and is the subject of this report.

2.0 SUMMARY

Evaluation of the NDA measurement results for the plutonium content of the first sixteen canisters of soil removed from the 216-Z-9 Crib Mining Facility has been completed. Results from the SAMS were found to be biased 25-55 percent high in relation to the SGSAS measurements. Within experimental error, this bias can be explained on the basis of differences in matrix density between the Los Alamos Scientific Laboratories (LASL)-prepared calibration standards and the mined soil, and to the inability of the SAMS instrument to correct for these differences. The major reason for this matrix density difference is due to the high moisture and organic content of the Z-9 surface soils when compared to the simulated standards.

Correction factors have been calculated which provide for adjustment of SAMS analytical results. These correction factors relate the theoretical attenuation correction factor of the sample canister to that of a representative simulated soil standard. These factors have been used to correct the SAMS results of the original sixteen canisters, the succeeding six canisters, and remeasurements of two of the original canister group. On the average, the corrected SAMS results agree with the SGSAS results to $+0.3 \pm 3.6$ grams at the 95% confidence level.

It is recommended that these derived correction factors be applied on a test basis for the remainder of the fifty canister analytical test, at which time a further evaluation will be performed. Concurrently, the use of alternate verification measurements, to further establish the reliability of SGSAS, will be investigated. Both NDA and destructive analytical techniques are being considered at this time. Until these alternate measurements are completed, it is suggested, based on previous experience with other sample types, that the SGSAS analytical results be used in making accountability, criticality, and disposal decisions related to these fifty canisters.

3.0 MEASUREMENT SYSTEMS AND CALIBRATION STANDARDS

Two gamma-ray measurement systems are currently being used for measurement of plutonium in 216-Z-9 soil canisters: (1) a Sodium-Iodide-detector based system located in the mining building and designated the Soil Assay Monitor System (SAMS), and (2) a sophisticated Ge(Li)-detector-based system located in Room 221A of the 234-5Z building and known as the Segmented Gamma Scan Assay System (SGSAS). Each system has been calibrated for the measurement of Z-9 soil by using simulated soil standards covering the plutonium range of 1-160 grams.

The Soil Assay Monitor System (SAMS) was developed and fabricated by the Development Engineering Department, ARHCO to provide a rapid and precise measurement of the plutonium content in a canister of soil. This is done by counting a selected gamma energy span (375-440-keV) for a preset time period. The count is then compared to a calibration curve determined from standard canisters (). The SAMS incorporates the following features to insure valid measurement data ().

1. A canister is placed within a lead shielded monitor assembly during counting to reduce background count to a very low level.
2. The canister is rotated on a turntable during counting to reduce differences caused by uneven mixing of plutonium in the soil.
3. The canister is weighed by a load cell for general information; the weight reading has no correlation with the plutonium measurement.

The Segmented Gamma-Scan Assay System (SGSAS) was fabricated by Canberra Industries and is based on design work originally performed at the Los Alamos Scientific Laboratories (reference 13). Briefly stated, the SGSAS has the following features:

1. A Ge(Li) detector is used because of its superior resolution (e.g., at 414 keV a high quality Ge(Li) will have a resolution of about 1.4 keV FWHM as compared to a high quality NaI with 40 keV FWHM). The obvious advantage is the ability to separate the peaks of interest from other neighboring gamma-ray peaks.
2. A gamma-ray transmission source, with emission energy close to that of the peak of interest, is located directly across from the detector and is attenuated by the presence of absorbing material in the sample. For the assay of plutonium at 414 keV, a ^{75}Se source (401 keV) is employed. (References 13, 17, addendum).
3. The sample is rotated (at about 12 rpm) to reduce count rate variation due to inhomogeneity in the radial location of the plutonium.

4. The detector is vertically collimated with lead bricks to view only a small, well-defined "slice" of the sample at any time. Each segment is treated as a separate counting sample and all segments are summed to obtain the total assay.
5. A computer is employed to control the mechanical scan apparatus, to collect and evaluate all count data from the assay and transmission peaks, to calculate attenuation correction factors, assay results, and error terms, and to prepare the assay report.

During assay of Z-9 soil canisters the SGSAS was set-up to view a one-inch segment during each 20 second counting period. The scan mechanism moves vertically during the count period as well as rotating, to produce a "helical" scan pattern. Fifteen segments were measured per canister although examination of the data printouts for both standard and sample canisters indicated that only 11 or 12 of the segments contained soil. The remainder were due to the bottom pedestal, to bagging material, and to the area near the top of the canister. Typical assay printouts are illustrated in Figures 1 and 2.

The Simulated Soil Calibration Standards were prepared by the CMB-1 Group (Analytical Chemistry) of the Los Alamos Scientific Laboratories, using specifications [REDACTED] provided by ARHCO and based on Research and Development Department analyses of 216-Z-9 covered trench soil samples (reference 16). Six standards, covering the 1-160 gram expected range of plutonium loadings were prepared using ARHCO-furnished sand, plastic bags, and metal containers. [REDACTED] Information [REDACTED], [REDACTED] is summarized in the following table:

TABLE I

PLUTONIUM CONTENTS OF NDA STANDARDS

<u>Standard</u>	<u>Pu Content*%/</u>
234-N-1-6	1.002 ± .002
235-N-2-6	1.997 ± .004
236-N-4-6	3.976 .009
237-N-11-6	10.57 .023
238-N-40-6	40.18 .088
239-N-160-6	160.2 .35

* Americium content of the plutonium adjusted to 2200 ppm as of 8-4-75.

+ ^{240}Pu atom-% calculated to be $5.95 \pm .05$ as of 7-24-75.

✓ All + values given at the 2σ level and include both systematic and random errors.

FIGURE 1: Typical Data Printout for Z-9 Soil
Standard (238-N-40-6)

THE PRESENT CALIBRATION IS:
NO,NO,NO,PU239,409.7KEV,NO,356KEV,Z-9 SAND,3101000, 15
LONG OR SHORT FORM? L
DO YOU NEED TO SUBTRACT TRANSMISSION INTERFERENCE? N

IDENTIFY THIS SAMPLE ? LASI SAND STD 238-N-40-6

ESTIMATE WT % PU-240: ? 6
HOW MANY TIMES DO YOU WANT THIS SAMPLE ASSAYED? 1
SEG # TRANSMISSION CORR FACTOR ASSAY PEAK

PU239

1	.4387	1.396	3.5
2	.0149	3.682	10.
3	.0158	3.636	13.8
4	.0188	3.502	15.5
5	.0128	3.806	11.7
6	.0208	3.423	14.7
7	.0204	3.436	12.2
8	.015	3.681	15.2
9	.0199	3.457	12.3
10	.0162	3.618	11.2
11	.0131	3.785	5.4
12	.1951	1.853	1.2
13	.6733	1.185	0
14	.8383	1.085	3.1
15	.5377	1.293	0

PU239	TOTAL GRAMS	35.47	% ERROR +/-	9.3
PU ASSAY	TOTAL GRAMS	37.96		

ENTER NAI ASSAY RESULT ?40.18
NAI RESULT SHOULD BE BETWEEN 32.96 AND 42.96 FOR AGREEMENT
THERE IS NO DISCREPANCY BETWEEN NAI AND SGSAS RESULTS

FIGURE 2: Typical Data Printout for Mined
Z-9 Soil Canister

THE PRESENT CALIBRATION IS:

NO,NO,NO,PU239,400.7KEV,NO,356KEV,Z-9 SAND,3101000, 15

LONG OR SHORT FORM? L

DO YOU NEED TO SUBTRACT TRANSMISSION INTERFERENCE? N

IDENTIFY THIS SAMPLE ? Z9C-76-08-0009

ESTIMATE WT % PU-240: ? 6

HOW MANY TIMES DO YOU WANT THIS SAMPLE ASSAYED? 1

SEG #	TRANSMISSION	CORR FACTOR	ASSAY PEAK
-------	--------------	-------------	------------

PU239

1	.4716	1.359	2.3
2	.0714	2.51	8.2
3	.0691	2.533	11.5
4	.0667	2.558	11.8
5	.0796	2.435	12.7
6	.0568	2.672	12.8
7	.0399	2.929	11.1
8	.0476	2.799	6.3
9	.0468	2.811	6.4
10	.0504	2.757	13.6
11	.0741	2.484	15
12	.1273	2.121	10.5
13	.5737	1.261	4.8
14	.8997	1.054	0
15	.6634	1.192	1.9

PU239	TOTAL GRAMS	35.19	% ERROR	+ -	8.5
PU ASSAY	TOTAL GRAMS	37.67			

ENTER NAI ASSAY RESULT ?54

NAI RESULT SHOULD BE BETWEEN 32.67 AND 42.67 FOR AGREEMENT

NAI AND SGSAS RESULTS DISAGREE -- CONTACT SUPERVISION

4.0 TEST DATA, CALCULATIONS, AND EVALUATION

From August 18, 1976 through August 26, 1976 sixteen canisters of plutonium-contaminated soil were removed from 216-Z-9 Crib. Upon removal from the packaging hood each canister was then placed into an outer storage canister and the lid taped on. Each canister was counted on the Soil Assay Monitor System (SAMS). Plutonium values were assigned to each canister, based on the observed net count and the posted calibration chart [REDACTED]. The canisters were then placed in fifty-five gallon steel waste barrels (maximum, three per barrel) and transported to the 234-5Z building NDA Counting Facility (Room 221A) for measurement with the Segmented Gamma Scan Assay System (SGSAS). All assay results are shown in Table II and plotted in Figure 3.

In each case, the SGSAS recognized the presence of a "discrepancy" (see footnote) and the computer program caused a second measurement to be made. Within the measurement uncertainty of the instrument, these second measurements agree with the original SGSAS results. As can be seen from Table II, the SAMS appears to be biased on the order of 25-55 percent high relative to the SGSAS. In virtually all cases, the SAMS assay results fall outside the established limit for measurement "agreement", which is about $\pm$ five grams in this concentration range, [REDACTED].

A check of each instrument's calibration was made by recounting one of the six simulated soil standards. The 40 gram standard was selected because it most closely matches the mined soil in plutonium content and the count rate is high enough that random counting errors would be small in comparison with the total count rate. These calibration check results are presented in Tables III and IV and show that, although low recoveries were obtained on both instruments, the response of these two instruments to the standard was virtually identical. No discrepancy of the magnitude observed for mined soil was evident. On the basis of these measurements, instrument problems were rejected as a possible cause for the discrepancies.

The SGSAS data printout was reviewed on a segment-by-segment basis to determine if there was any physical reason for such a large bias between the two instruments. Of particular interest were the measured transmission ratios observed for the mined soil samples and those observed for the simulated soil standards. As can be seen from Table V, there is a significant difference between the two material types in this physical property.

(text continues on page 15)

"discrepancy" - defined in reference 8 'as a difference between the NaI assay and SGSAS assay values of more than 1.41 times the computer calculated SGSAS error (95% confidence level) or 5 grams, whichever is greater.'

TABLE IICOMPARATIVE ASSAY RESULTS: NaI SOIL MONITOR vs SGSAS

<u>Date</u>	<u>Sample*</u>	<u>Soil Monitor gms Pu</u>	<u>SGSAS gms Pu</u>	<u>SM-SGSAS SGSAS</u> x 100
8-18-76	Z9C-76-08-0001	59	42.6	+38.5 %
			41.6	41.8
	Z9C-76-08-0003	68	46.7	45.6
			44.0	54.5
8-23-76	Z9C-76-08-0004	88	59.9	46.9
			59.2	48.6
	Z9C-76-08-0005	72	51.0	41.2
			51.4	40.1
	Z9C-76-08-0006	49	39.0	25.6
			39.0	25.6
8-24-76	Z9C-76-08-0007	35	23.9	46.4
			22.9	52.8
	Z9C-76-08-0008	79	54.7	44.4
			49.0	61.2
	Z9C-76-08-0009	54	37.7	43.2
			39.0	38.5
	Z9C-76-08-0010	53	37.3	42.1
			34.2	55.0
8-26-76	Z9C-76-08-0011	48	34.5	39.1
			31.9	50.5
	Z9C-76-08-0012	31.5	21.8	44.5
			23.2	35.8
	Z9C-76-08-0013	43	31.1	38.3
			30.0	43.3
	Z9C-76-08-0014	25.5	18.9	34.9
			18.0	41.7
	Z9C-76-08-0015	23	17.7	29.9
			19.0	21.1
	Z9C-76-08-0016	54	35.6	51.7
			37.7	43.2
	Z9C-76-08-0017	44	32.6	35.0
			31.4	40.1

* Sample Z9C-76-08-0002 contained uncontaminated sand and had been prepared for demonstrations of the glovebox bagout and loading procedures.

FIGURE 3: Original Data: SgsAS assay results vs SAMS assay results

TABLE IIISIMULATED SOIL STANDARD (238-N-40-6) RESULTS by SGSAS

<u>Date</u>	<u>SGSAS Assay gms Pu (1)</u>	<u>Percent Recovery (2)</u>
8-17-76	37.0 $\pm$ 0.2	92.0 %
8-18-76	38.6 $\pm$ 1.4	96.1
8-20-76	38.0	94.6
8-23-76	37.6 $\pm$ 0.1	93.6
8-24-76	36.4 $\pm$ 1.6	90.6
8-25-76	35.8 $\pm$ 0.5	89.0
8-26-76	36.5	90.8
Overall	37.1 $\pm$ 2.1	92.5 %

(1) 95% confidence limit shown except where only a single measurement was made.

(2) 238-N-40-6 Standard plutonium value = 40.18 grams

TABLE IVSIMULATED SOIL STANDARD (238-N-40-6) RESULTS by SAMS

<u>Date</u>	<u>SAMS Assay gms Pu (1)</u>	<u>Percent Recovery</u>
8-19-76	38.0 $\pm$ 2.24	94.6 %
	38.0 $\pm$ 2.24	94.6
	37.5 $\pm$ 2.22	93.3
	38.0 $\pm$ 2.24	94.6
	37.5 $\pm$ 2.22	93.3
Overall	37.8	94.1 %

(1) 95% confidence limits

TABLE V

TRANSMISSION OF 401 keV ^{75}Se GAMMA-RAYS
THROUGH SIMULATED AND ACTUAL Z-9 SOILS

<u>Canister or Standard</u>	<u>Measured Percent Transmission</u>
Std 1 (1 gm)	1.7 %
2 (2 gm)	1.8
3 (4 gm)	1.6
4 (11 gm)	1.8
5 (40 gm)	2.0
6 (160 gm)	4.6
Canister -0001	7.2 %
-0003	9.5
-0004	11.8
-0005	10.1
-0006	6.7
-0007	6.5
-0008	10.4
-0009	6.6
-0010	9.4
-0011	7.3
-0012	4.6
-0013	7.0
-0014	6.0
-0015	4.2
-0016	8.7
-0017	8.7
Canister -0018	5.3
-0019	2.8
-0020	4.8
-0021 (rocks)	4.9
-0022	5.5
-0023	3.8

"Effective" transmission calculated using the
relationship:

$$\hat{T} = \frac{\sum T_i \times C_i}{\sum C_i} \quad \text{where: } T_i = \text{transmission through the } i\text{th segment}$$

C_i = measured ^{239}Pu counts in the i th segment

These transmission values provided a "key" to resolution of the observed measurement bias, in that they demonstrated some fundamental relationships and constraints of gamma-ray assay.

T, or transmission, is the fraction of gamma-rays from the source (^{75}Se at 401 keV) which pass through the sample with no change in energy or direction. This fraction is energy-dependent but is virtually the same at 401 keV and at 414 keV (the energy at which ^{239}Pu has a major gamma-ray peak) so no further correction is necessary. The transmission measurement is, therefore, a good estimate of the matrix attenuation of the plutonium gamma-rays being emitted from the sample and can be used to develop correction factors to calculate what this emission rate would be in the absence of matrix (ideal case). Because of the close proximity of the two gamma-ray peaks, less than 13 keV separating them, a high resolution or Ge(Li) detector is required to make the measurements.

While transmission is a property that can be measured for any sample, and correction factors calculated, it can also be predicted for any material if some other information is available. In order to do so, it is necessary to know the following three properties of the material to be measured:

- the mass attenuation coefficient of the material, or μ , in cm^2/gram .
- the density, ρ , in grams/cm^3 .
- the diameter of the sample, D, in cm.

Unfortunately, the mass attenuation coefficient, which is specific for each energy, is also specific for the exact material to be measured--whether it be wood, sand, metal, concrete, paper, etc. (reference 16). The composition of Z-9 sand is too varied with respect to material composition (sand, rocks, silt, water, organics, etc.) to make more than a rough guess as to what the mass attenuation coefficient should be. By making transmission measurements of the matrix material in the first sixteen Z-9 soil canisters we had all the information available to make an estimate as to the relationship between the material density and its transmission properties. The fundamental relationship (reference 13) described above can be expressed in the following mathematical form:

$$T = e^{-\mu\rho D}$$

or alternatively, $-\ln T = \mu \cdot \rho \cdot D$

For the sake of expediency it was assumed that μ is a constant throughout the Z-9 crib soil (an assumption not fully tested by the limited number of samples assayed to date). D, the diameter of the canister, is also a constant and has been measured at 19.685 cm. Therefore, the only

terms of the equation which would be expected to vary from canister to canister are the transmission and the density, with $\mu \cdot D$ the slope of the line relating the two variables. This relationship has been plotted in Figure 4 and the least-squares slope and intercept of the dashed line are calculated as 1.585 and 0.505, respectively. The linear correlation coefficient is 0.94, indicating a reasonably good fit to the line by the experimental points.

To carry fundamentals a little further, an accepted (references 13 and 17) attenuation correction factor expression for cylindrical samples is given by the following equation:

$$CF = \frac{k \cdot \mu' \cdot D}{1 - e^{-k \cdot \mu' \cdot D}} \quad \mu' = \mu \cdot \rho = \text{linear attenuation coefficient, cm}^{-1}$$

where k is a constant of value ~ 0.8 . A first-order approximation of the exact far-field form for the cylinder gives $k = \pi/4 = 0.785$, whereas empirical and numerical studies indicate $k = 0.82$ is better. For $k = 0.82$, CF is within 1-1/2% of the correct value for $0.01 \leq T \leq 1$ and within $\pm 3\%$ for $0.001 \leq T \leq 1$.

This is recognized as being only an approximate expression, but it works quite well even when the detector is only one diameter from the edge of the cylindrical sample. The correction factor equation can be rewritten in terms of T :

$$CF = \frac{k \ln T}{T^k - 1}$$

Figure 5 shows a plot of CF vs T for slab and cylindrical samples. For T greater than 0.2, CF is less than 2.0 and does not change rapidly. This is a favorable range for assay work, but unfortunately we do not have much control over the characteristics of the Z-9 soil (except the diameter of the can), and we are in the position of working with a material having a T consistently less than 0.2. In this region CF rises rapidly and the possibility of error increases. Measured transmissions become less accurate when T is less than 10%.

Figure 4 could now be used to predict the average transmission to be expected from a canister of Z-9 soil from a measurement of its density. This required that each and every canister be measured for volume and weight of soil so that the density of the soil could be calculated. A representative, clean, canister was measured with water to determine its volume. A crease in the canister wall, approximately 1-1/2 inches below the top edge was chosen as a reference point. The volume of the inner soil canister was determined to be 9.1 liters at this level.*

* Estimates of density for the first six canisters were made based on the apparent depth of soil seen by the SGSAS transmission measurement and, as such, are probably subject to more than normal error. All subsequent canisters were filled to the depth indicated above.

FIGURE 4: SGSAS-MEASURED TRANSMISSION VALUES
vs CALCULATED Z-9 SOIL DENSITIES

FIGURE 5: Far-field correction factor for cylindrical and slab-type samples as functions of transmission. (Reference 17)

Special Services and Product Handling (Z-9) was instructed to fill all subsequent canisters to this level ([REDACTED] Weight measurements were made with the load cell installed in the SAMS and are estimated to be accurate to ± 300 gram (net weight of 10-16 Kg).

Attenuation correction factors were then calculated for all sample canisters, using the weight and volume measurements indicated to calculate T, and then using this T to calculate CF, as given in the previous equation. Density, transmission, and CF values were also calculated for the 40 gram simulated soil standard.

Since the calibration chart prepared for the SAMS was constructed using measurements of the simulated soil standards, it is obvious that a certain amount of correction factor is "built-in". Therefore, it was only necessary to determine the ratio of correction factors between the mined soil and the simulated soil standard (i.e. measurement of any standard on the SAMS should give the correct answer without further correction because the ratio of "sample" CF to "standard" CF would be 1.0). Table VI shows the calculated attenuation correction factors for the Z-9 soil canisters and the 40 gram standard, as well as the CF-ratio (hereafter known as the FACTOR). Figure 6 is a plot of calculated FACTORS for the expected range of canister net weights (triangular points). The black points represent the FACTORS calculated for each canister when the measured transmission values are used.

Table VII is a compilation of the corrected SAMS assay results when the derived FACTOR is used. A comparison is made to the SGSAS assay results and the assay difference shown. On the average, the agreement between the corrected SAMS and SGSAS is $+0.3 \pm 3.6$ grams at the 95% confidence level. Table VIII shows the results obtained on canisters -0018 to -0023 when the appropriate FACTOR was used. These canisters were not involved in the original data evaluation and can be considered as "independent" measurements. Correlation between the two instruments is shown by figure 7 and it is worthwhile to note that not one of the corrected SAMS assay values falls outside the "agreement" range (± 5 grams).

In conclusion, it is felt that the entire observed bias between the SAMS and the SGSAS assay results is accounted for by a consideration of the matrix density differences between the mined soil samples and the simulated soil standards. Without further physical examination of the mined soil one cannot completely explain the reason for these differences except to assume that the presence of excessive moisture and organic compounds contributes to the low material densities observed. Since the mined soil is only passed through a one-inch screen it is also reasonable to assume that there are many voids or air pockets in the canisters. The simulated soil standards exhibit higher density because they were prepared as free-flowing soil standards containing a minimum of liquid (1 to 4 weight percent) by the Los Alamos Scientific Laboratory. This was necessitated by various constraints against the shipment

(text continues on page 24)

TABLE VIATTENUATION CORRECTION FACTORS
FOR Z-9 CANISTERS AND SOIL
STANDARDS (FROM DENSITY MEASUREMENTS)

<u>Material</u>	<u>Net Weight</u>	<u>Volume</u>	<u>CF</u>	<u>FACTOR</u>
40 gm std	16.48 kg	8.31	3.148	.942
-0001	9.7	7.34	2.420	.724
-0003	6.9	6.57	2.139	.640
-0004	9.5	9.28	2.116	.633
-0005	11.6	8.89	2.400	.718
-0006	12.0	8.31	2.550	.763
-0007	12.2	9.08	2.443	.731
-0008	10.1	9.08	2.203	.659
-0009	12.0	9.08	2.420	.724
-0010	12.1	9.08	2.430	.727
-0011	11.9	9.08	2.406	.720
-0012	15.5	9.08	2.838	.849
-0013	12.2	9.08	2.443	.731
-0014	14.1	9.08	2.667	.798
-0015	15.4	9.08	2.824	.845
-0016	10.5	9.08	2.246	.672
-0017	10.9	9.08	2.293	.686
-0018	13.3	9.08	2.570	.769
-0019	16.2	9.08	2.921	.874
-0020	14.8	9.08	2.751	.823
-0021	13.3	9.08	2.570	.769
-0022	14.1	9.08	2.667	.798
-0023	15.3	9.08	2.811	.841

Figure 6: Calculated Correction FACTORS for different densities of Z-9 Soil (triangular points);

Correction FACTORS for specific Z-9 canisters calculated from measured transmissions (black points)

TABLE VIICOMPARATIVE ASSAY RESULTS: CORRECTED NaI SOIL MONITOR vs SGSAS

Sample	Soil Monitor gms Pu	FACTOR	Corrected gms Pu	SGSAS gms Pu	Difference
Z9C-76-08-0001	59 gms	.724	42.7 gms	42.1 gms	+ 0.6
Z9C-76-08-0003	68	.640	43.5	45.3	- 1.8
Z9C-76-08-0004	88	.633	55.7	59.6	- 3.9
Z9C-76-08-0005	72	.718	51.7	51.2	+ 0.5
Z9C-76-08-0006	49	.763	37.4	39.0	- 1.6
Z9C-76-08-0007	35	.731	25.6	23.4	+ 2.2
Z9C-76-08-0008	79	.659	52.1	51.9	+ 0.2
Z9C-76-08-0009	54	.724	39.1	38.3	+ 0.8
Z9C-76-08-0010	53	.727	38.5	35.8	+ 2.7
Z9C-76-08-0011	48	.720	34.6	33.2	+ 1.4
Z9C-76-08-0012	31.5	.849	26.7	22.5	+ 4.2
Z9C-76-08-0013	43	.731	31.4	30.5	+ 0.9
Z9C-76-08-0014	25.5	.798	20.3	18.5	+ 1.8
Z9C-76-08-0015	23	.845	19.4	18.4	+ 1.0
Z9C-76-08-0016	54	.672	36.3	36.6	- 0.3
Z9C-76-08-0017	44	.686	30.2	32.0	- 1.8
			585.2 gms	578.3 gms	+ 6.9 gms

TABLE VIIICOMPARATIVE ASSAY RESULTS: CANISTERS MEASURED AFTER CORRECTION
FACTOR TABLE IN USE

Z9C-76-08-0018	25.5 gms	.769	19.6 gms	21.3 gms	- 1.7 gms
Z9C-76-08-0019	6.1	.874	5.3	4.5	+ 0.8
Z9C-76-08-0020	21	.823	17.3	16.1	+ 1.2
Z9C-76-08-0021*	1.8	.769	1.4	2.3	- 0.9
Z9C-76-08-0022	31	.798	24.7	24.5	+ 0.2
Z9C-76-08-0023	15.2	.841	12.8	12.8	0
			81.1 gms	81.5 gms	- 0.4 gm

*Rock canister: material not passing through 1" screen

Density-Corrected NaI Soil Monitor- grams Plutonium

FIGURE 7 : Corrected Data: SGSAS assay results vs Corrected-SAMS Assay results

○ = Canisters -0001, -0003 to -0017

● = Canisters -0018 to -0023

of plutonium nitrate solution standards. The density of dry sand is between 2.0 and 2.2 grams/cm³ and the soil standards exhibit densities consistent with their low moisture content.

It does not appear that the preparation of additional, lower density standards will allow us to assay the Z-9 soil to any better accuracy and precision than is possible using the derived FACTORS to compensate for variations in canister density (i.e. net weight). While the preparation of "representative" calibration standards is a time-honored and much-used (and occasionally abused) technique, it does not seem appropriate when the sample composition seems to vary as much as the Z-9 soil. The use of an attenuation correction factor derived from transmission measurements, as discussed in this report, is believed to be a much more versatile technique and one which does not require the preparation, documentation, and storage of a large inventory of representative standards. The preparation of several standards containing no plutonium, but varying only in matrix density, could be of value in verifying the observed correlation between canister density and transmission.

Phase II of the analytical plan is concerned with further dual measurements of the Z-9 soil canisters (up to canister 50) and it is recommended that the derived FACTORS be applied during the remainder of the test. A further evaluation will be made at the conclusion of measurements on the first fifty canisters and the FACTOR calculation table will be revised as necessary to reflect this additional data.

Concurrent with further SGSAS-SAMS data evaluation, several other analytical techniques are being investigated to further establish the reliability of the SGSAS. Briefly, the various approaches that are being considered for further work are as follows:

- Repackaging of a "typical" Z-9 soil canister into approximately two kilogram (1.2 liter) quantities for subsequent measurement.
- SGSAS measurement of the smaller packages.
- SAMS counting of the smaller packages.
- Calorimetry of the smaller packages.
- Neutron Well Coincidence Counting (NWCC) of the smaller packages by the Hanford Engineering Development Laboratory (HEDL) to determine the spontaneous fission isotopes (²³⁸Pu, ²⁴⁰Pu, and ²⁴²Pu).
- Wet chemical analysis of the Z-9 soil.
- Preparation of a "working" standard by the addition of a known quantity of plutonium to a previously measured soil canister.

5.0 REFERENCES

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6.0 APPENDIX

CORRECTION FACTORS FOR USE WITH THE Z-9 NAI SOIL MONITOR

<u>Net Weight (Kg)</u>	<u>Factor</u>	<u>Net Weight (Kg)</u>	<u>Factor</u>
9.0	0.623	15.0	0.830
9.2	.629	15.2	.838
9.4	.636	15.4	.845
9.6	.642	15.6	.852
9.8	.649	15.8	.860
10.0	.656	16.0	.867
10.2	.662	16.2	.874
10.4	.669	16.4	.882
10.6	.676	16.6	.889
10.8	.683	16.8	.897
11.0	.689	17.0	.904
11.2	.696	17.2	.912
11.4	.703	17.4	.919
11.6	.710	17.6	.927
11.8	.717	17.8	.934
12.0	.724	18.0	.942
12.2	.731	18.2	.950
12.4	.738	18.4	.957
12.6	.744	18.6	.965
12.8	.752	18.8	.973
13.0	.759	19.0	.980
13.2	.766	19.2	.988
13.4	.773	19.4	.996
13.6	.780	19.6	1.003
13.8	.787	19.8	1.011
14.0	.794	20.0	1.019
14.2	.801		
14.4	.809		
14.6	.816		
14.8	.823		

-
- 1) A constant fill volume of 9.08 liters is assumed in the preparation of these factors. This is equivalent to a fill depth of 11-3/4 inches in the inner canister.
 - 2) Tare weight of empty canisters and inner plastic bag should be determined daily.

TABLE IX

ADDITIONAL COUNTING DATA
ON SELECTED CANISTERS

<u>SAMPLE</u>	<u>NET WEIGHT (Kg)</u>	<u>FACTOR</u>	<u>SAMS (gms)</u>	<u>CORR. SAMS</u>	<u>SGSAS gms Pu</u>
Z9C-76-08-0009	12.0	.724	54	39.1	37.7
					39.0
	12.0	.724	53	38.4	42.5
	12.0	.724	52	37.6	37.8
	11.9	.720	52	37.5	41.0
	12.2	.731	52	38.0	37.7
mean	12.02		52.6	38.1	39.3
variance s^2	0.012		0.8	0.43	4.12
Z9C-76-08-0011	11.9	.720	48	34.6	34.5
					31.9
	11.4	.703	47	33.0	33.7
	11.6	.710	47	33.4	32.4
	11.9	.720	47	33.8	33.8
	11.6	.710	46	32.7	32.0
mean	11.68		47.0	33.5	33.05
variance s^2	0.047		0.5	0.55	1.19

7.0 ADDENDUM

As this report was nearing the final stages of preparation the author received his current copy of Nuclear Materials Management, the Journal of the INMM (of which he is a member), which includes an article by J.L. Parker and T.D. Reilly of LASL dealing with exactly the type of problem encountered in the foregoing report (reference 17). The following information is quoted directly from the article:

OUTLINE OF METHODS USED TO DETERMINE CORRECTION FACTORS

Representative Standards. The oldest and perhaps still most used (and abused) method is that of avoiding the issue by using representative standards....This procedure will produce good results if (and only if) the unknowns and standards are sufficiently similar that the same concentration of assay material in each gives rise to the same sample μ and, therefore, to the same correction factor. In other words, at the same concentration of assay material, the exact same fraction of gamma rays must escape from both sample and standard. This method is only applicable in cases where the nature and composition of the assay samples are well known and unvarying

Computation from Knowledge of Composition. A second method exploits previous knowledge of the chemical composition, mass, and shape to compute μ of the sample from which in turn the correction factor can then be computed. Sufficient prior knowledge to compute the sample μ does not necessarily imply that the assay result is known in advance....When highest accuracy is not necessary and the required knowledge is available, this approach is useful.

Gamma-Ray Intensity Ratios. Another method of determining attenuation corrections, more discussed than actually used, involves measuring the relative intensity of two gamma rays of different energy emitted by the material under assay....some prior knowledge of the nature of the sample is required....the method has proved useful in particular cases, and it does have the potential for raising a warning flag when the assumption of reasonable uniformity is not met.

Transmission Method. A fourth and most general method of obtaining the attenuation correction involves experimental determination of the sample μ directly by measuring the transmission through the sample of a beam of gamma rays from an external source. This method requires no knowledge of the chemical composition of the sample, just the basic assumptions on uniformity and particle size. As such,

it is the only practical way to proceed in the "black box" cases and is often the preferred method even when some knowledge of the sample composition is available, especially if the best obtainable accuracy is desired.

* * * * *

"Just a word about experimental standards is in order. In principle, a single standard will suffice if the proper selection of $CF(T)$ has been made, but clearly, it is prudent to have two or more standards spanning the expected range of values of T . It should be emphasized that the standards need not have the same or even similar chemical composition as the unknowns inasmuch as the CF is dependent only upon the transmission value, sample shape, and assay geometry."