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**USE OF THE KNOWN-M METHOD FOR  
NONDESTRUCTIVE ASSAY OF PLUTONIUM SCRAP**

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**ABSTRACT**

Plutonium scrap from another Department of Energy site is to be converted at Savannah River Site (SRS) to a form for permanent storage. For accountability and criticality safety, the material must be measured at SRS, and handling restrictions require assay in 9975 shipping drums. A Multiplicity Neutron Counter is available to perform the measurements, but requires about 12 hours per assay, too long to support the measurement schedule. The assay time has been reduced to 2 hours by use of the Known-M method, the first known routine application of Known-M. The approach involves expression of the multiplication in terms of the effective <sup>239</sup>Pu mass and a quadratic polynomial. Because only a few measured values of multiplication were available, values from Monte Carlo neutron transport calculations (using code MCNP) were used. Because the scrap cans have variable fill heights and fill height affects multiplication, an algorithm to correct the effective <sup>239</sup>Pu mass values for that effect was

developed. Testing of the Known-M calibration with limited data suggests a 2-sigma uncertainty of about 5%. Drums can contain one or two individual scrap cans, and an algorithm for measuring the combined plutonium content in two cans was developed. The Known-M assay calculations will be performed off line using a spreadsheet.

## INTRODUCTION

Sand, slag and crucible (SS&C) material containing plutonium is being shipped to SRS for conversion into a form suitable for long-term storage. It is necessary to measure the plutonium content of the material upon receipt at SRS for purposes of nuclear material accountability and criticality safety. Because some of the material has lumps, neutron measurement techniques are more suitable for the measurement than are gamma ray techniques. A neutron multiplicity counter that can assay up to 200-liter drums is available in the receipt facility for performing the assays, but its low neutron counting efficiency (~17%) and the high fraction of ( $\alpha$ , n) neutrons from the SS&C material would require multiplicity count times of 12 hours or longer. Only a small fraction of the incoming drums could be assayed with such long count times, so a measurement technique allowing shorter count times was needed. Because of the consistency of the material composition and its predictable geometry (can dimensions and fill height), we decided to implement the Known-M method<sup>1</sup>. Although only limited assay data are currently available, the method appears to work well for SS&C material. This is believed to be the first use of the Known-M method in routine facility nondestructive measurements.

## SUMMARY

The Known-M method is being implemented at SRS for determination of the plutonium content of sand, slag, and crucible material in cans packaged in 9975 shipping drums. The material can be packaged in a single can or in two stacked cans in the drum, but the determination is of the total plutonium mass in the drum, not that in each individual can. The Known-M calculation is performed using a spreadsheet and requires the following input data: the shipping or inventory value of plutonium mass in each individual can, the fill height of each can, the Singles and Doubles (Totals and Reals) count rates for the drum, and their associated uncertainties. In addition, the plutonium isotope mass fractions and the relative <sup>241</sup>Am mass are required. The count rates and uncertainties are determined using the Neutron Multiplicity Counter in the "Rates Only" mode. For a 100 g sample, a count time of two hours results in a 2-sigma statistical uncertainty of about 5%. Uncertainties in the fill heights can dominate the propagated measurement uncertainty.

## DISCUSSION

**Method** The Known-M method involves solution of the following equations<sup>1,2</sup>:

$$M = C_0 + C_1 \cdot (m_{239})_{\text{eff}} + C_2 \cdot (m_{239})_{\text{eff}}^2 \quad (1)$$

where  $M$  is the Leakage Multiplication,  
 $(m_{239})_{\text{eff}}$  is the effective <sup>239</sup>Pu mass, and  
 $C_0$ ,  $C_1$ , and  $C_2$  are coefficients determined by experiment or calculation.

$$\alpha = \{ M / [ D / (S \rho_0) - K ( M^2 - M ) ] \} - 1 \quad (2)$$

where  $\alpha$  is the ratio of ( $\alpha$ , n) neutrons to spontaneous fission neutrons,  
 $D$  is the Doubles (Reals) count rate,  
 $S$  is the Singles (Totals) count rate,

$$\rho_0 = \varepsilon f_D v_{s2} / (2 v_{s1}),$$

$$K = v_{i2} v_{s1} / [v_{s2} (v_{s1} - 1)] \cong 2.17,$$

$\varepsilon$  is the neutron counter efficiency,  
 $f_D$  is the doubles gate fraction, and  
 $v_{ij}, v_{sj}$  are the  $j$ th moments of the induced and  
spontaneous fission neutron multiplicities, respectively.

$$(m_{240})_{\text{eff}} = S / [473.5 \varepsilon v_{s1} M (\alpha + 1)] \quad (3)$$

For any neutron-based measurement, the isotopic composition of the items to be assayed must be known. In this case, the equations are given in terms of the effective  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  masses to more accurately account for induced fission (equation (4) below) and spontaneous fission (equation (5) below) effects. These are generally defined as follows<sup>1</sup>:

$$(m_{239})_{\text{eff}} = m_{\text{Pu}} [f_{239} + 0.786 f_{238} + 0.515 f_{240} + 1.414 f_{241} + 0.545 f_{\text{Am}241}] \quad (4)$$

$$(m_{240})_{\text{eff}} = m_{\text{Pu}} [2.52 f_{238} + f_{240} + 1.68 f_{242}] \quad (5)$$

where  $m_{\text{Pu}}$  is the calculated plutonium elemental mass, and  
 $f_n$  is the isotopic mass fraction of  $^n\text{Pu}$  or  $^{241}\text{Am}$ .

Except for equation (1), the above equations are the same as the equations for a neutron multiplicity calculation<sup>2</sup>. For a multiplicity calculation, the first equation is a cubic equation in the parameters used above in equations (2) and (3), plus the Triples count rate and Triples gate fraction. The cubic equation is solved for  $M$ , and then in succession equations (2) and (3) are solved, and, using the known isotopic fractions, the plutonium mass is thereby obtained. But neutron multiplicity counting was not suitable for SS&C material and the available neutron counter. First, the SS&C material produces a large number of  $(\alpha, n)$  neutrons, manifest in a relatively large value of  $\alpha$ , ranging from about 5 to about 35. (The two uses of  $\alpha$  here may be momentarily confusing:  $(\alpha, n)$  refers to interactions of  $\alpha$ -particles emitted from plutonium and other radioisotopes with low- $A$  nuclei, and symbol  $\alpha$  defined in equation (2) is the ratio of the number of  $(\alpha, n)$  neutrons to the number of spontaneous fission neutrons.) Material generally assayed by multiplicity counting has  $\alpha \sim 1$ . Second, the efficiency for counting Triples events is  $\varepsilon^3$ , and the relatively low efficiency of the available neutron counter ( $\varepsilon \approx 17\%$ ) makes counting Triples very inefficient. As a result of these two conditions (large  $\alpha$  and small  $\varepsilon$ ), a multiplicity measurement using the available neutron counter requires count times of 12 hours or more to obtain satisfactory statistical accuracy.

The Known- $M$  method is applicable if the material to be assayed is consistent enough in composition and geometry to allow expression of the multiplication  $M$  as in equation (1). The Triples count rate and its uncertainty are not needed. In this case, the plutonium mass, the result of the overall calculation, is needed in equation (1), the very start of the calculation. In fact, the Known- $M$  equations must be solved by iteration. Fortunately, the convergence behavior of the series of equations is such that the iteration function of the Microsoft Excel™ spreadsheet can be used. To avoid the divergence that occurred in many cases, an algorithm to “encourage” convergence was devised, and the cell formula for that purpose is discussed below.

The first step in developing a Known-M calibration is to determine the coefficients  $C_0$ ,  $C_1$ , and  $C_2$  in equation (1). These coefficients are obtained by fitting a quadratic function to leakage multiplication values for items containing different effective  $^{239}\text{Pu}$  mass. The multiplication data can be obtained experimentally, or they can be obtained by Monte Carlo calculations. Note that  $C_0$  must be very close to unity, since  $M$  approaches 1 as the quantity of fissile material approaches 0.

In the equations above,  $\epsilon$  and  $f_D$  are the “calibration coefficients.” Although  $\epsilon$  and  $f_D$  can be determined for a given detector using a  $^{252}\text{Cf}$  source, the determination is based on the energy of  $^{252}\text{Cf}$  neutrons, and the specific neutron energies produced by the material being assayed may cause small changes in  $\epsilon$  and  $f_D$ . The appropriate values for these parameters are determined by adjusting them to minimize the differences between the “known” contents of and assay results for standard cans packed in shipping drums and counted in the neutron counter.

**Height Correction** The SS&C material has a fairly consistent composition, and the cans containing the material are all the same size. However, the fill fraction of the cans varies from less than 10% to as much as 100% because of shipping limits on the plutonium content of a single can and process requirements. Because the multiplication  $M$  is affected strongly by differences in fill height, a correction factor for this effect was developed. The fill height correction factor  $\Psi(h\%)$  for a can filled to  $h\%$  ( $h\% = 100$  for a completely full can) is defined as follows:

$$\Psi(h\%) = [ \text{Chord}(100\%) / \text{Chord}(h\%) ]^2 \quad (6)$$

$$\begin{aligned} \text{Chord}(h\%) &= \text{Average dimension of the can} \\ &= 2r [ (2/3) ( G^3 - H^3 ) + GH + \ln(G + H) ] / ( 2H + 1 ) \end{aligned} \quad (7)$$

$$\begin{aligned} \text{where } H &= [h / (2r)] (h\%/100), \\ G &= ( H^2 + 1 )^{1/2}, \\ r &\text{ is the interior radius of the can, and} \\ h &\text{ is the interior height of the can.} \end{aligned}$$

This correction factor  $\Psi(h\%)$  becomes a component of the effective  $^{239}\text{Pu}$  mass for calculation of  $M$ . Equation (4) is thus replaced by equation (4a). Note that for a completely full can,  $\Psi(100\%) = 1$ , and the correction factor has no effect on the effective mass.

$$(m_{239})_{\text{eff}} = m_{\text{Pu}} [ f_{239} + 0.786 f_{238} + 0.515 f_{240} + 1.414 f_{241} + 0.545 f_{\text{Am}241} ] \Psi(h\%) \quad (4a)$$

The equation for the Chord was obtained by determining the average length of a line segment bounded by the cylinder and passing through its center. This was obtained by evaluating the integral:

$$\text{Chord} = ( 1 / A_S ) \int_S \lambda \, dS \quad (8)$$

where  $\lambda$  is a bounded line segment through both the cylinder center and  $dS$ ,

$A_S$  is the surface area of the cylinder, and  
The integral is over the surface of the cylinder.

Other functions were tested, and the Chord was tested raised to powers other than 2, but the function shown above gave by far the best geometry correction.

**Fill Height Determination** In addition to the multiplicity neutron counter, a gamma isotopic system is also available at the SRS receipts facility. This instrument is designed to scan a drum along its axis, determining the transmission of  $^{137}\text{Cs}$  gamma rays through the drum at elevations  $\frac{1}{2}$  inch apart. The resulting plot is then used by the operator to determine the elevation at which to position the drum so that a SS&C can is directly in front of the detector so that a gamma ray spectrum for isotopic analysis can be taken. The same plot shows the profile of the SS&C material in the can or cans in the drum, and can be used to determine the approximate height of the material. If there are cases for which the scan plot is inconclusive as to the fill height(s), the fill height(s) can be determined from a radiograph.

**Calibration Standards** Calibration standards were obtained by characterizing cans of actual SS&C material using a gamma isotopic fraction determination and calorimetry. The isotopic mass fractions determined by gamma analysis were in good agreement with the expected values for the material type. The calorimetry measurements of each can were performed several times each on each of two entirely different SRS calorimeters. The 1-sigma uncertainty of the assigned plutonium masses for these standard cans is thought to be about 1% for plutonium masses greater than 50 g. A total of seven standard cans were characterized this way. These cans had fill height fractions ranging from 14% to 87%. Each single standard can packaged in a 9975 shipping drum was counted in the neutron counter multiple times, and six different pair combinations of cans in 9975 drums were also counted several times each. In all, neutron count rate data for thirteen drum and can configurations were obtained and used for the Known-M calibration.

**Determination of  $C_0$ ,  $C_1$ , and  $C_2$**  Multiplicity vs.  $(m_{239})_{\text{eff}}$  values over the range of plutonium mass allowed in a single can were needed to fit a quadratic curve and define the  $\{C_i\}$ . Because only seven standards were available and the multiplications determined by 12-hour multiplicity neutron counts had relatively large uncertainties, it was necessary to obtain the necessary data by computation. The Monte Carlo code MCNP was used to model a single can in a 9965 drum. The 9965 is very similar to the 9975, the major difference being the 9975 has a cylinder of  $\frac{1}{2}$ -inch-thick lead enclosing the primary containment vessel which contains the can. The  $\{C_i\}$  were determined for 9965 drums, but appear to work equally well for the 9975 drums. Because of time constraints and the success obtained with the 9965 data, MCNP calculations have not been done for the 9975 drum configuration.

MCNP calculations were done for 12 cases: Four plutonium mass values, each mass value at a low, a medium, and a high fill height. For a good test of the fill height correction, 11 different fill height fractions were used: 5, 10, 15, 20, 30, 50 (two cases), 55, 75, 80, 85, and 90%. Figure 1 shows the

multiplication results from the Monte Carlo calculations for the 12 cases plotted without corrections for the fill height. The data show that, for a fixed quantity of plutonium in a can, the multiplication of the material increases as the fill height decreases. They also show the necessity of a fill height correction.

**Figure 1. Multiplication for Different Fill Heights Without Height Correction**

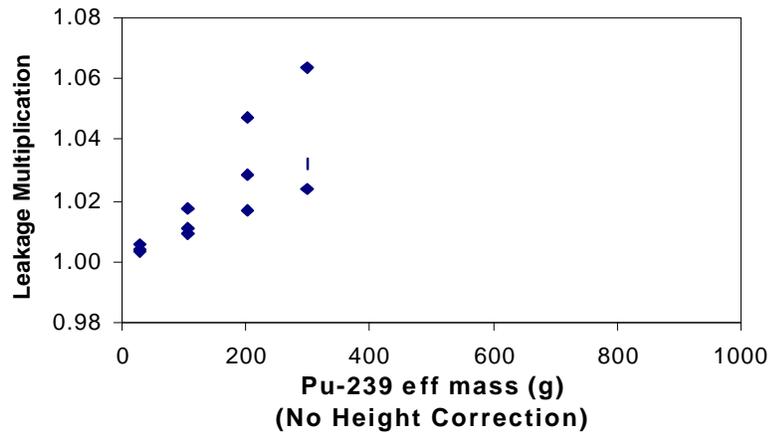
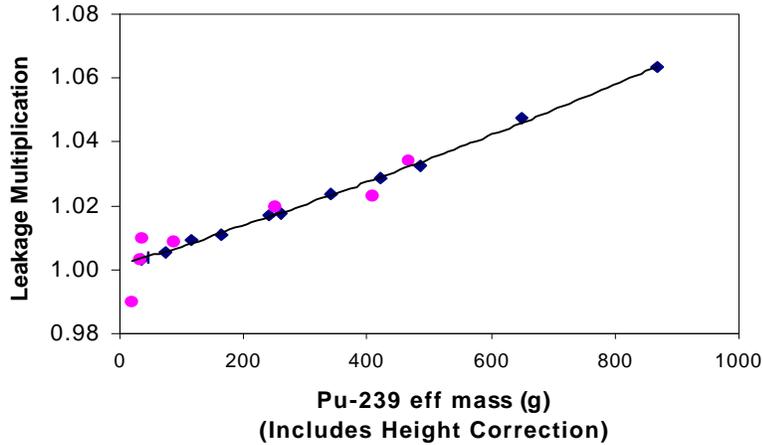


Figure 2 (below) shows the same calculation results, but with the  $^{239}\text{Pu}$  effective masses including the fill height correction factor. The 12 points now form a curve that can be fit well by a quadratic function, as seen in the figure. This graph indicates that the geometry correction factor works very well. The circular data points are multiplication values obtained from multiplicity counts on each standard can. These experimental values are in reasonable agreement with the calculated values, which provides validation of the results of the Monte Carlo calculations. The coefficients of the quadratic curve determined for the multiplication equation are:

$$\begin{aligned} C_0 &= 1.00113 \\ C_1 &= 5.42 \times 10^{-5} \\ C_2 &= 3.69 \times 10^{-9} \end{aligned}$$

**Figure 2. Multiplication for Different Fill Heights With Height Correction**



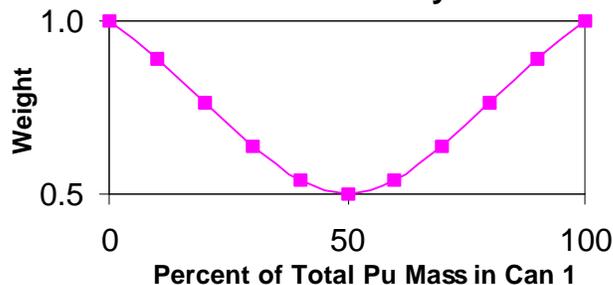
**Can Combination Algorithm** When two SS&C cans are packaged in a 9975 drum, one can is on top of the other, and a steel plate separates the two cans. Because of the plate, the rolled seals of the can tops and bottoms, and the empty space generally above the SS&C material in the bottom can, the SS&C material in the two cans is somewhat decoupled with respect to neutron multiplication. In fact, for the purposes of this calculation, it is assumed that the SS&C in the two cans is completely decoupled, so that the multiplication in each can is unaffected by the SS&C material in the other. Now, if essentially all the plutonium in a drum is in a single can, the multiplication for the calculation for that drum is the multiplication for the can. If, on the other hand, the plutonium mass is equally divided between two cans in a drum, the multiplication for that drum calculation is obtained using  $\frac{1}{2}$  the total plutonium in the drum; that is, the multiplication of the two cans will be about equal (depending upon their relative fill heights), and the presence of either can does not affect the multiplication of the other. These considerations suggest the following expression for the effective  $^{239}\text{Pu}$  mass when two cans are packaged in a 9975:

$$(m_{239})_{\text{eff}} = m_{\text{Pu}} \left[ 1 - \left( \frac{m_1 m_2}{m_1^2 + m_2^2} \right) \right] \times \left[ \frac{(m_1 \Psi(h_1\%) + m_2 \Psi(h_2\%))}{(m_1 + m_2)} \right] (f_{239})_{\text{eff}} \quad (9)$$

where  $m_1$  and  $m_2$  are the shipper values of the plutonium masses in the cans, and  $(f_{239})_{\text{eff}}$  is the effective  $^{239}\text{Pu}$  isotope mass fraction (equation (4) with  $m_{\text{Pu}} = 1$ ).

The effective  $^{239}\text{Pu}$  mass  $(m_{239})_{\text{eff}}$  calculated by equation (9) is used in equation (1) to determine the multiplication  $M$  for the drum. The first factor in equation (9) is the plutonium mass from the last calculation iteration. The second factor is the weighting function  $\left[ 1 - \left( \frac{m_1 m_2}{m_1^2 + m_2^2} \right) \right]$  the behavior of which is shown in Figure 3 as a function of the fraction of plutonium in Can 1. The third factor is the combined fill height correction factor obtained by weighting the respective can fill height correction factors by the shipper values of the plutonium masses in the cans. The last factor is the effective  $^{239}\text{Pu}$  mass fraction. The shipper or inventory masses are needed to evaluate these functions, but only the relative values are used in the calculations.

**Figure 3. Weighting Function for Two-Can Assays**



**Convergence Algorithm** The iterative calculation for many drums diverged rather than converging, and so an algorithm was developed to “encourage” convergence: An upper bound UB for the assay plutonium mass value is specified, say UB = 500. This upper bound should be somewhat larger than the plutonium mass any drum is expected to contain. If a calculated plutonium mass is greater than zero and less than or equal to the value UB, then the plutonium mass for the next iteration is the average of that value and the mass result of the last iteration. If the calculated plutonium mass is outside the interval [0, UB], then the plutonium mass for the next iteration is obtained by multiplying a random number between 0 and 1 times UB. In an Excel™ spreadsheet, a formula for this algorithm is as follows:

$$=IF(AND([CalcVal]>=0,[CalcVal]<=[UB]), +([PrevVal]+[CalcVal])/2, +RAND()*[UB])$$

where [CalcVal] is the address of the cell containing the value calculated this iteration,  
 [PrevVal] is the address of the cell containing the value calculated last iteration, and  
 [UB] is the address of the cell containing the upper bound UB.

**Uncertainty Calculation** The input variables having dominant effects on the uncertainty of the calculation are the Doubles rate D and the fill height fractions  $h_1\%$  and  $h_2\%$ . The Singles uncertainty is so small as to be inconsequential, and the uncertainty effect of the isotopic fractions is assumed to be negligible. Because the solution of the Known-M equations is iterative, a calculation of the uncertainty in closed form is not possible. Instead, the Known-M computation spreadsheet includes iterative calculations for  $\Delta D$ ,  $\Delta h_1\%$ , and  $\Delta h_2\%$  in addition to each mass calculation, and each of these three additional calculations has the appropriate variable perturbed by  $\frac{1}{2}$  the uncertainty in the variable:  $D + \Delta D/2$ ,  $h_1\% + \Delta h_1\%/2$ , and  $h_2\% + \Delta h_2\%/2$ . The resultant mass from each calculation corresponds to the perturbed input variable, and so taking the difference between the mass calculation value and a perturbed result and then doubling the difference gives the effect of the uncertainty for the particular variable on the calculated mass. Since the three variables  $\Delta D$ ,  $\Delta h_1\%$ , and  $\Delta h_2\%$  are independent, they can be combined in quadrature. This uncertainty computation process can be represented as follows, where  $\Delta m_{Pu}$  is the uncertainty in  $m_{Pu}$ :

$$\Delta m_{Pu} = \{ ( 2 m_{Pu}( D + \Delta D/2 ) - m_{Pu} )^2 + ( 2 m_{Pu}(h_1\% + \Delta h_1\%/2) - m_{Pu} )^2 + ( 2 m_{Pu}(h_2\% + \Delta h_2\%/2) - m_{Pu} )^2 \}^{1/2} \quad (10)$$

**Calibration Results** The calibration coefficients  $\epsilon$  and  $f_D$  were determined by adjusting them to minimize the sum of the weighted differences between the calculated plutonium masses and the standard mass values. The Solver feature of the Excel™ Spreadsheet was used to perform the minimization. The minimized quantity is defined by:

$$\sum_i | (m_{Pu})_i / (\Delta m_{Pu})_i |.$$

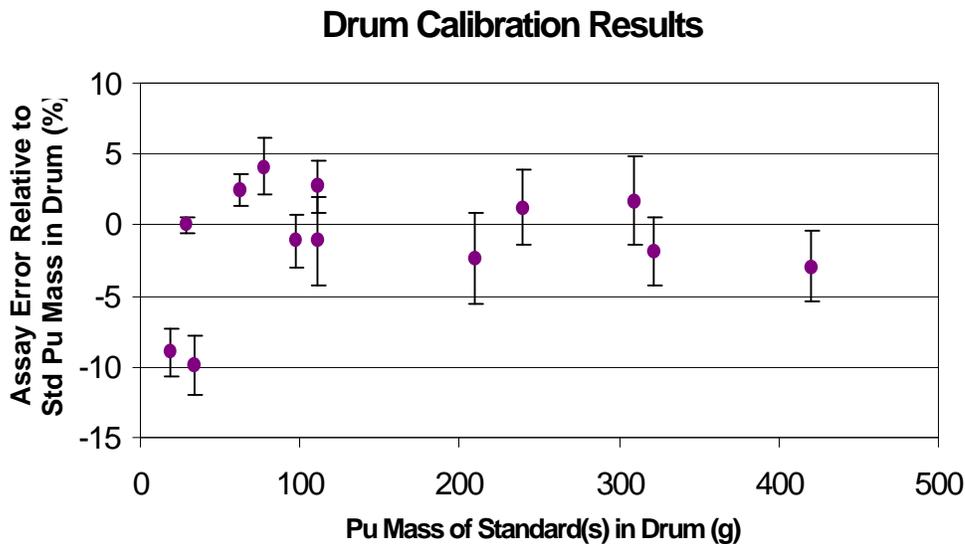


Figure 4 shows the calibration results. The assay uncertainties are 1-sigma values. The standard data include thirteen points, two of which were excluded from the calibration fit because their calculated masses differed by about 10% from the standard mass values, whereas the agreement for the other eleven points was much better. The justification for excluding the two points is that the calorimetry results for small mass values ( $< \sim 35$  g) have much larger uncertainties than those for higher masses. The two points excluded from the fit are shown in Figure 4.

An estimate of the measurement uncertainty was obtained by treating the relative differences between the calculated masses and the standards as a sample of a population having a normal distribution and calculating its mean and standard deviation. The resulting values are  $\mu = 0.26\%$  and  $\sigma = 2.33\%$  for assay parameters  $\epsilon = 0.1550$  and  $f_D = 0.5620$ . As in the case for the calibration fit, the two poorer-fitting data points were excluded from the calculation of  $\mu$  and  $\sigma$ . The quality of agreement between the Known-M and standard masses is very good. However, more assay data are needed to fully validate the calibration. These data will be available once receipt of the offsite SS&C material gets underway.

**Assay Spreadsheet** Figure 5 shows the first page of the four-page Known-M Computation Spreadsheet. This spreadsheet performs all calculations to determine the plutonium masses in 9975 Shipping Drums and their uncertainties. It also provides the M and  $\alpha$  values, as well as the relative difference between the reference plutonium mass (from shipper or inventory) and the measured mass for each drum.

## Computation Spreadsheet for Known-M NDA Method

Material Type: Repackaged SS&C assayed in 9975 drums

Detector/Counter ID: NMC using standard isotopics

<b>Detector Characterization Parameters</b>				<b>Mult vs (m239)eff</b>			
v11 = 3.163	v11 = 2.154	$\epsilon = 0.1550$		C0 = 1.00113			
v12 = 8.24	v12 = 3.789	$f_d = 0.5620$		C1 = 5.420E-05			
	$\rho_0 = 0.0766$	$C_1 = 2.1657$		C2 = 3.690E-09			
<b>IF Prob wrt <sup>239</sup>Pu</b>		<b>SF Prob wrt <sup>238</sup>Pu</b>		<b>SRS Can Parameters</b>			
a238 = 0.786	Pu238/Pu240 = 2.52		Diameter = 10.24 (cm) rf can				
a240 = 0.515	Pu242/Pu240 = 1.68		Height = 13.59 (cm) rf can				
a241 = 1.414			Chord = 13.46 cm @ 100% Fill Ht				
a Am241 = 0.545			Ht Correction = 1.00 cm				
			Ht Uncertainty $\sigma_{ht}$ = 0.50 cm				
<b>Intermediate Values</b>							
			h/d		1.33		
			G		1.66		

Drum ID	Date	Can#	Can 1/Can 2	Count Rate Data		Mult	Ratio	Result		Asy vs Inv			
#	Analyst	Ident	Inv mPu	Scan Ht (in)	Tots/Reals	s(Tots/Reals)	M	$\alpha$	mPu (g)	s(mPu)%	Diff%		
1	Drum 1	06/24/99	1	Std A	111.1	0.75	31133.38	0.59	1.0157	28.76	109.87	3.15	-1.10
		RST	2		0	0	163.98	1.06					
2	Drum 2	06/24/99	1	Std B	29	2.7	70358.87	1.35	1.0211	29.39	241.92	2.62	1.22
		RST	2	Std C	210	1.9	433.25	3.81					
3	Drum 3	06/24/99	1	Std E	77.69	3.16	34629.05	0.90	1.0061	31.20	114.05	1.85	2.75
		RST	2	Std F	33.3	4.64	118.00	1.89					

Figure 5. First page of the Spreadsheet used to perform the Known-M calculations. Lightly shaded areas are input fields.

### CONCLUSIONS

Based on the limited data available for the calibration, the Known-M method implemented at SRS works very well for SS&C material, with an expected measurement uncertainty of about 5% 2-sigma. More extensive comparison data are needed to fully validate the method, and those data will be available once the offsite SS&C material begins to arrive at SRS. Use of Known-M in general should provide improved accuracy compared to traditional use of a neutron coincidence counter, for which 2-sigma measurement uncertainties can exceed 20%.

### ACKNOWLEDGEMENTS

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