



**ENVIRONMENTAL
MANAGEMENT**



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**Performance Testing and Validation Plan for
HMS4 Quantitative Gamma Measurements,
K-25/K-27 D&D Project,
East Tennessee Technology Park,
Oak Ridge, Tennessee**

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**Performance Testing and Validation Plan for
HMS4 Quantitative Gamma Measurements,
K-25/K-27 D&D Project,
East Tennessee Technology Park,
Oak Ridge, Tennessee**

Date Issued - April 2008

Prepared for the
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Office of Environmental Management

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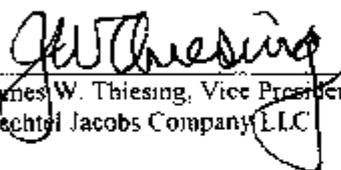
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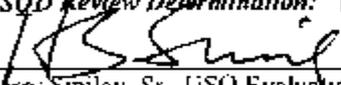
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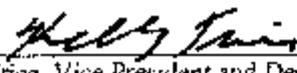

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ACRONYMS

BJC	Bechtel Jacobs Company LLC
DQO	Data Quality Objective
GGH	Generalized Geometry Holdup
eV	Electron Volts
FOV	Field of View
HMS4	Holdup Measurement System 4
LANL	Los Alamos National Laboratory
LLD	Lower Level of Detection
MDA	Minimum Detectable Amount
NDA	Nondestructive Assay
PTVP	Performance Testing and Validation Plan
ROI	Region of Interest
RSD	Relative Standard Deviation
TMU	Total Measurement Uncertainty
VPD	Vent, Purge, and Drain

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1. HMS4 SYSTEM DESCRIPTION

1.1 DESCRIPTION OF MEASUREMENT SYSTEM

The Holdup Measurement System 4 (HMS4) is a portable thallium activated sodium iodide (NaI(Tl)) gamma ray energy spectrometer that, when properly calibrated, is able to make quantifiable assessment of U-235 holdup in the presence of other uranium isotopes and prevailing background radiation.

The use and calibration of the HMS4 is based upon the methodologies defined by Russo in LA-14206, (Russo 2005), where detection efficiency determination protocols are defined (called Generalized Geometry Holdup [GGH]). The GGH methodology together with attenuation correction algorithms and other modeling parameters are combined in the HMS4 software package to provide a comprehensive tool for conducting in situ gamma-ray measurements. The fundamental principles of these capabilities will be discussed in more detail in Sect. 2.2.

Figure 1.1 shows a schematic diagram of the NaI(Tl) detection element and its housing for the HMS4 system. Figure 1.2 shows a schematic diagram of the spectrometer primary system components. The detector element is a 1.0 inch diameter by 0.5 inch thick scintillation crystal of NaI(Tl). Incident radiation photons from the source material of interest, and for that matter, non source generated interfering background radiation interacting in the crystal, results in the generation of photons in the crystal in the near visible region. A photomultiplier tube is utilized to convert the deposited photon energy into an output current pulse with an output proportional to the energy input.

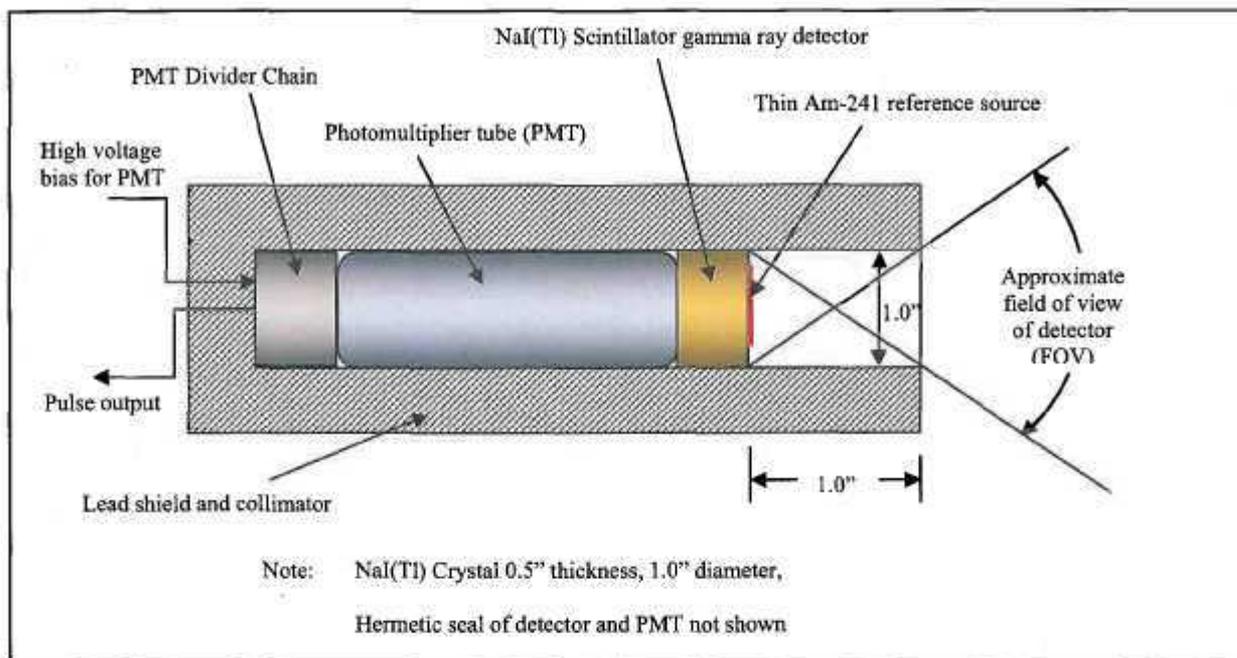


Fig. 1.1. Shielded NaI(Tl) detector utilized for the HMS4.

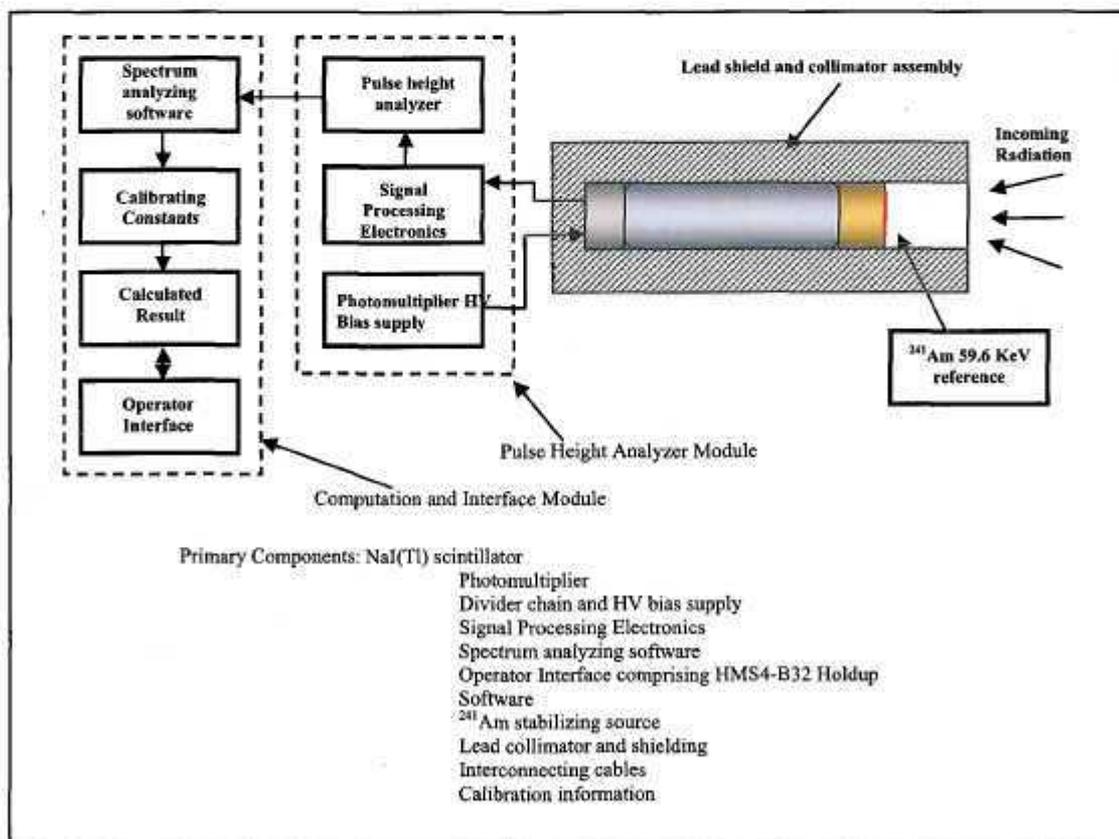


Fig. 1.2. Conceptual diagram of HMS4 scintillation spectrometer system.

The output current pulse is processed into a form and shape suitable for pulse height analysis. This pulse output is then received by a pulse height analyzer where its height is measured and stored. Input pulses are collected for a set period of time so that an energy spectrum can be formed and displayed.

An Am-241 source is positioned on the scintillator to provide a constant reference source of gamma-rays at 60 keV. This provides a constant full energy peak that does not interfere with the observation of U-235 events. This peak provides a data quality check and an electronic gain stabilization signal for the electronics system.

Figure 1.3 shows an energy spectrum observed from an HMS4, showing the presence of the stabilizing Am-241 source and U-235. A region of interest (ROI) can be selected so that pulses (counts or events) falling in this region may be attributed to the source material of interest. A higher energy ROI (sometimes known as window) is set to quantify the Compton continuum from the 1001 keV gamma of U-238 in the holdup material on which the 186 keV U-235 gamma is "riding."

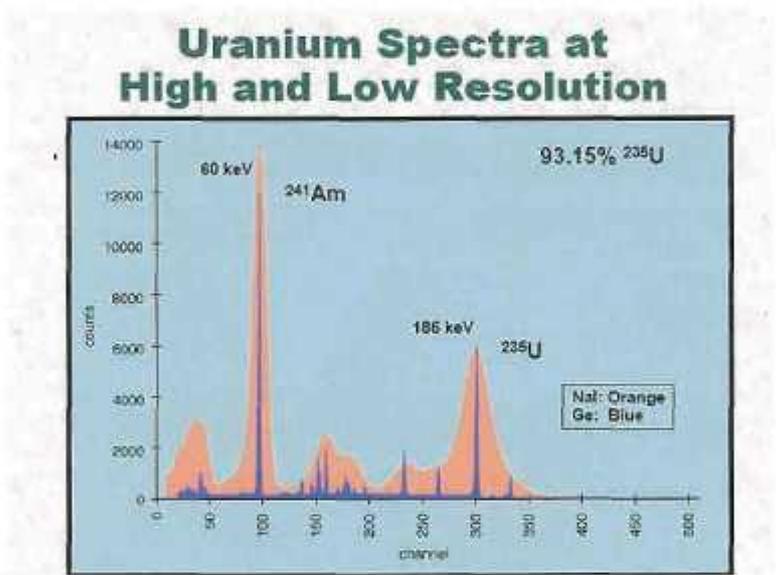


Fig. 1.3. Energy spectrum observed with an HMS4 for U-235.
(Note the 60 keV peak resulting from the Am-241 reference source. For comparative purposes, an Energy Spectrum is also shown using a Ge Gamma Ray Detector.)

As with all gamma ray spectra, there are multiple phenomena occurring that result in apparent spectral peaks which can be due to scatter and other origins that complicate the analysis. For example, U-235 and its associated isotopes, because of its photons interacting in the source and matrix material, will generate fluorescing characteristic X-rays resulting in a series of spectral peaks in the observed spectrum (Fig.1.4). Setting ROIs selects only the peaks of interest for analysis.

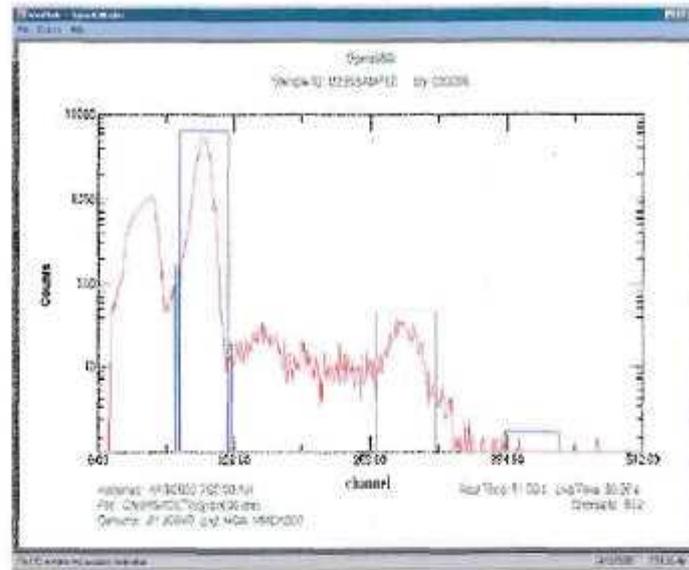


Fig. 1.4. Energy spectrum with set regions of interest for analysis.

1.2 EFFICIENCY DETERMINATION

1.2.1 Defining Absolute Efficiency of Detection Ψ

The use of the HMS4 will result in a full energy peak in the energy spectrum representing U-235, as shown in Fig. 1.3. Measurement of the area of this peak will result in a determined value of the peak intensity either as counts per unit time or counts collected over time T. If the relationship is known between the counts measured and the source—detector geometry for that measurement—then the amount of U-235 can be calculated.

Ψ (conversion factor for detector efficiency (calibration), source self-attenuation, matrix and geometric attenuation, and finite point or line source dimensions) is calculated as:

$$\Psi = A/B = K * CF_{sa} * CF_{fw} * CF_{ma} \quad (1.1)$$

where,

A = Net (background subtracted) counts per unit time measured in the U-235 186 keV full energy peak observed with the HMS4 spectrometer

B = Grams of U-235 in the source material being measured

K = Detector calibration coefficient

CF_{sa} = Correction factor for source self-attenuation

CF_{fw} = Correction factor for finite source width (point or line)

CF_{ma} = Correction factor for matrix attenuation caused by materials between the source and the detector.

Figure 1.5 is a schematic diagram of the HMS4 measuring a source containing U-235. Note that this is a conceptual diagram. The source material emits photons in all directions (4π). Only a certain fraction travel towards the detector as defined by its solid angle. Of this fraction, only a portion will be detected and registered as collected events in the energy spectrum (Fig. 1.3). The remainder are eliminated by several phenomena, including source self-attenuation, detector collimation, intervening materials, and detector efficiency. Where the full energy of the gamma ray is deposited in the detector, then a full energy peak event is registered. A partial deposition of energy results in the event being registered in a lower region of the energy spectrum.

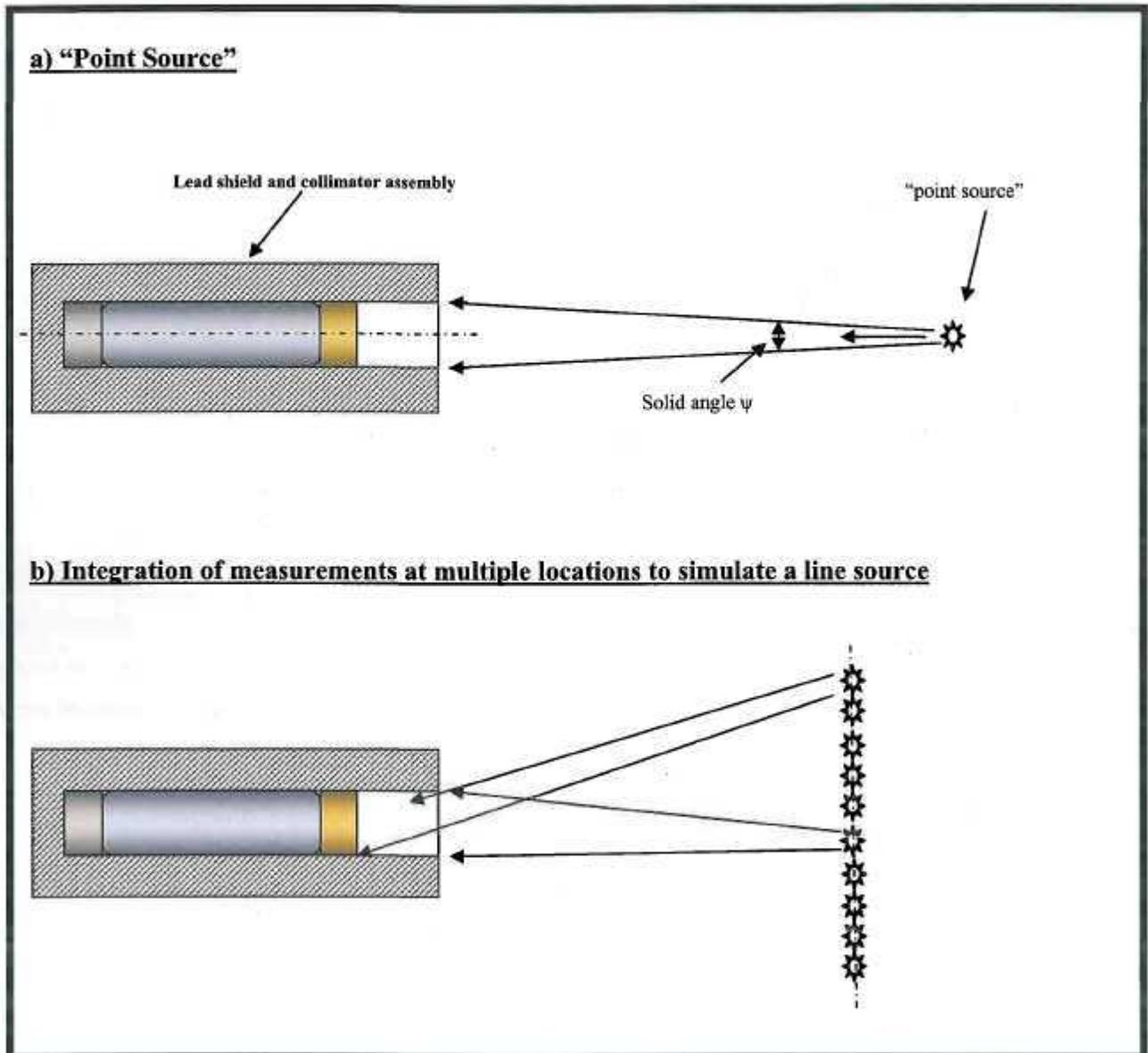


Fig. 1.5. Measurement geometry for absolute efficiency of detection.

Determination of the source activity requires that the source geometry must be accommodated along with accounting for all absorbing media between the source material and the detector. Additionally, interfering background counts from scatter and background sources must also be subtracted from the peak ROI.

1.2.2 Point Source and Extended Source Measurement

Several references from Russo (Russo 2005), and a Holdup Workshop (ORNL 2007) are listed to provide an excellent assessment for geometric (GGH determination) source measurements and most other aspects of this measurement.

Any extended source can be closely approximated by successively measuring a point source at multiple locations to simulate that extended geometry. This methodology has been adapted at Los Alamos National Laboratory (LANL) to GGH measurement with HMS4 to closely approximate the three pertinent measurement geometries. The HMS4 measures those geometries that approximate to a point, a line (whose lateral extent completely covers the detector field of view [FOV]), and an area source (whose lateral extent completely covers the detector FOV [i.e., large piping, duct walls, and flooring]). The fundamental principles of these capabilities will be discussed in more detail in Sect. 2.2.

For this work, a calibration method is used where a known small geometry source is measured in multiple locations to effectively cover the extended geometry of interest.

The detector efficiency, K_p , for a point source is measured by counting a known point source placed on the extended centerline of the detector a known distance from the detector face.

The detector efficiency, K_l , for a line source is measured by counting a known point source placed at several different locations along a line perpendicular to the extended detector centerline, intersecting the centerline at a known distance from the detector face. This produces a "radial response curve" that is integrated over the detector FOV to simulate the detector response to a line source.

The detector efficiency, K_a , for an area source is calculated by integrating the line source response as the line is rotated 180° to simulate an area source completely filling the detector FOV.

2. THEORY OF OPERATION

2.1 DETECTION AND MEASUREMENT CONCEPT

2.1.1 Detection Theory

Gamma rays emitted from radionuclides have characteristic energies of emission that are fixed, irrespective of any external criteria. Each radionuclide emits a unique energy (or range of energies) whose precise measurement can allow the identification of the presence of that radionuclide. In the case of the uranium isotope series of interest, each disintegration results in the emission of an alpha particle immediately followed by one or more monoenergetic photons.

If the response of the detector is proportional to the energy deposited in its volume, then the detector has an energy measurement capability and can be useful as an energy spectrometer. The crystal NaI(Tl) is optically transparent. A gamma ray interacting in the crystal transfers some, or all of its energy to an electron (Compton interaction or photoelectric effect). This energetic electron then travels through the crystal lattice, losing energy to the crystal lattice through lattice excitation, the generation of more electrons and the generation of Bremsstrahlung X-rays. These are typically reabsorbed into the crystal lattice. The crystal de-excites through the emission of numbers of blue visible region photons.

The light is generated as a light "pulse" whose intensity is proportional to the energy deposited in the crystal. This light is detected and amplified by use of a device called a photo multiplier tube (PMT).

Figure 2.1 shows how a typical scintillation crystal and a photomultiplier are coupled together to make a complete detection element. The photomultiplier requires a voltage distribution so that the avalanche of electrons can be accelerated down the tube to provide an output current pulse. This device is very sensitive to changes in voltage and as a consequence requires care in use to ensure its gain stability.

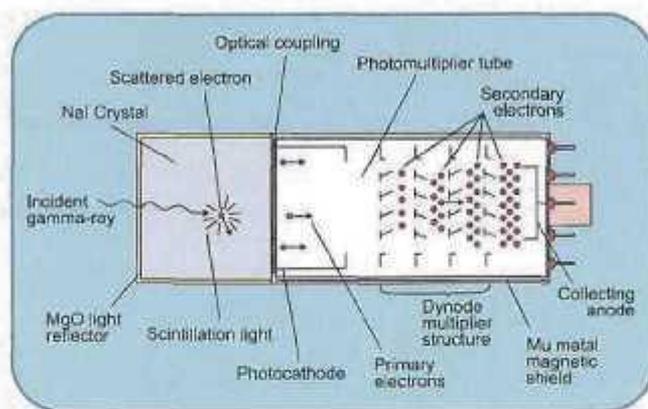


Fig. 2.1. Diagram of a typical scintillator and photomultiplier assembly.

The photomultiplier coupling to the detection crystal is sealed in a light tight hermetic housing that also protects the anhydrous NaI(Tl) crystal from water absorption. The photocathode is arranged to be close to the crystal to maximize optical coupling efficiency.

Typically, 300 to 500 electron volts (eV) are required to eject one photoelectron from the photocathode. If the energy deposited from the radiation interaction in the crystal is 186 keV then 370 to 620 photo electrons are generated. This range has a statistical variance which is intrinsic to the scintillation process. Other losses can occur where electrons escape and other energies also escape because of coupling efficiencies, system, detection geometries and crystal qualities. This is the limiting factor of its ability to resolve differing energy gamma rays.

Several phenomena have been discussed so far that influence the detector full energy peak efficiency. These include:

- Self absorption in the source material
- Absorption in materials between the source and the detector
- Detector collimator
- Incomplete absorption of the incident photon energy
- Source-detector geometry

Each contribution can be complex, particularly when the source material may have an extended or large geometry. This is the subject of calibration discussions later in this text.

From the above discussion, gamma ray energies are resolved with a moderate energy resolution to show a peak of the type shown in Fig. 2.2 below. This peak is situated upon background events not associated with the energy of interest and therefore must be subtracted. This figure accommodates this Compton continuum (from the 1001 keV gamma of U-238) but does not accommodate changes in the slope of the Compton continuum.

In Fig. 2.2, two ROIs have been defined, each containing the same number of channels. The first is the region of the 186 keV peak (denoted "P" in the diagram, and referred to as ROI 4 in HMS4 operational literature), and a region above the peak containing the Compton continuum from interactions of higher energy gamma rays, primarily the 1001 keV gamma-ray of U-238 (denoted "B" in the diagram, and referred to as ROI 5 in HMS4 operational literature.)

In the K-25 Bldg., a background measurement will exhibit a similar structure, since there exists noticeable amounts of U-238 and U-235 gamma-rays in the background radiation coming from other, non-targeted components. The background spectrum is obtained by moving the detector so that its directional vector is unchanged while removing the target component from the detector FOV. Identical ROIs are defined for the background spectrum as for the foreground spectrum. To facilitate further discussion, define the following parameters:

x_4 = total counts in the foreground plus background spectrum in ROI 4.

x_5 = total counts in the foreground plus background spectrum in ROI 5.

x_{B4} = total counts in the background spectrum in ROI 4.

x_{B5} = total counts in the background spectrum in ROI 5.

Net Peak-Area Analysis

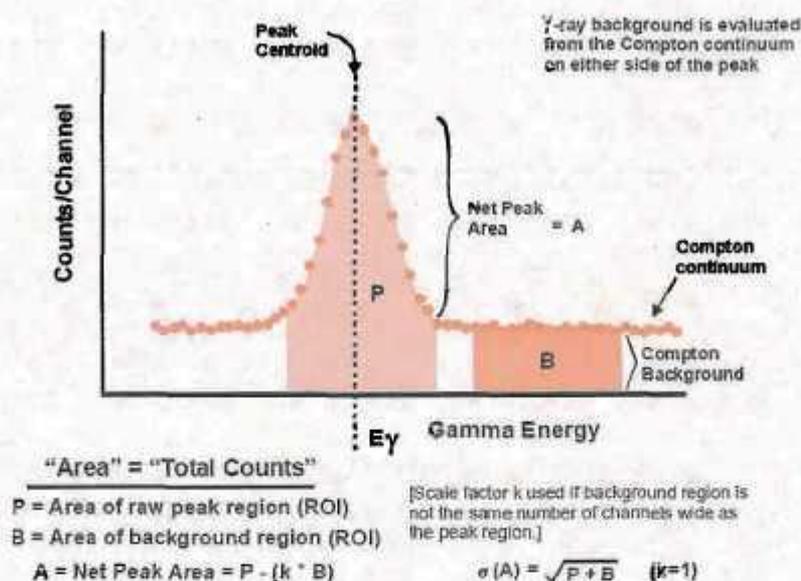


Fig. 2.2. Observation of 186 keV peak and subtraction of the background continuum.

To get the net counts from the target source material, it is necessary to subtract two components from the ROI 4 counts in the foreground plus background measurement. The first is the contribution of background to the 186 keV photo-peak of U-235 ($x_{B4} - x_{B5}$). The second is the Compton continuum from higher energy gamma-rays on which the 186 keV photo-peak is riding (x_5). Note that this continuum is also subtracted in the background spectrum before subtracting the background from the foreground plus background to avoid double-subtraction of that contribution to the Compton continuum.

The net counts in the ROI defining the 186 keV U-235 peak is thus:

$$x_{\text{net}} = x_4 - (x_{B4} - x_{B5}) - x_5 \quad (2.1)$$

If there is attenuating material between the target source and the detector, the background must be corrected for the attenuation it would have encountered before reaching the detector, as discussed in Sect. 2.3.

2.2 WIDTH LIMITATIONS FOR POINT AND LINE SOURCES

Russo (Russo 2005) develops finite point diameter and finite line with correction factors discussed in Sect. 2.3 of this document. Her development discusses width limitations for these source configurations based on the radial response curve for the HMS4 instrument. An example of such a response curve is shown in Fig. 4.2. Russo recommends that the diameter of a finite point source or the width of a finite line source be no greater than the full width at half maximum of the radial response, since the efficiency of the detector-collimator configuration decreases rapidly at radial locations outside this limit. If there is a non-uniformity in the point or line source outside the stated limit, the detector will not quantify this portion of the response very well, and could grossly understate or overstate the mass of such a deposit.

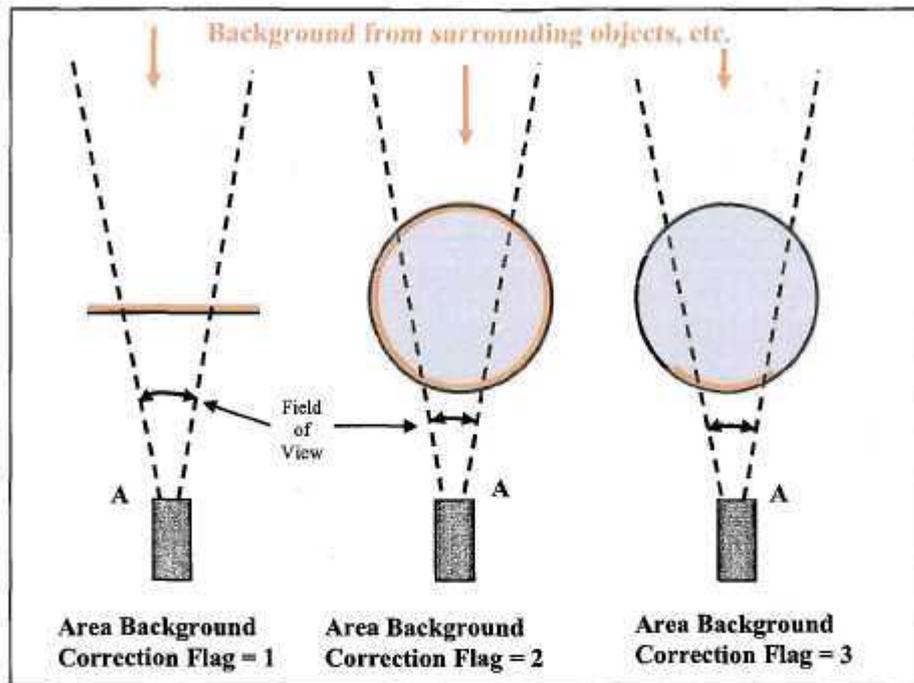
For the specific geometry of the HMS4 detection system, the full width of the response at half maximum can be approximated from Fig. 4.3 as 40% of the detector FOV. Limits on point and line source finite dimensions will be stated as 40% of the detector FOV for practical operational reasons throughout this document.

2.3 GENERALIZED GEOMETRY HOLDUP APPROXIMATION CONCEPTS

The use of HMS4 must result in meaningful assessment of the presence and quantification of the U-235 source materials. This specifically requires that the Total Measurement Uncertainty (TMU) (see Sect. 5.4) be properly determined for the reported value of the assessed material. Because the size and distribution of holdup deposits are unknown or poorly known it is useful to approximate these factors in a way which can be accounted for, given the known properties of radiation detection discussed previously. The use of simple, defined geometric models is utilized to approximate the unknown size and distribution of the holdup deposits. The HMS4 detector is then calibrated in order to determine the efficiency of the detector to detect the desired radionuclide for the simple, defined geometric model.

The HMS4 point, line, and area calculations assume that the basic source distributions are defined as:

- A point source simulates a small, disk-shaped deposit that is completely inside the detector's FOV. A point source should occupy 40% or less of the diameter of the detector FOV.
- A line (simulating small pipe deposition) source is a narrow source that extends beyond the detector's FOV in one dimension. A line source fills the FOV from one side to the other while generally occupying 40% or less in the other dimension.
- An area source (simulating large duct, floor, etc.) extends beyond the detector FOV in all directions and is assumed to be uniform in mass distribution. Area sources are subdivided into three types. These types and their corresponding background correction flags are shown in Fig. 2.3.



$$cCR = (A - (B/t)) * t$$

$$cCR = ((A - (B/t))/2) * t$$

$$cCR = (A - (B/tt)) * t$$

where,

A = Measured Count Rate

B = Background

cCR = Count Rate corrected for
source distribution,
material attenuation, and
background

— = Material Deposit

t = Transmission correction factor (one wall thickness)

tt = Transmission correction factor (wall thickness*2)

Fig. 2.3. Area background correction types.

Due to the exponential nature of material attenuation, use of HMS4 in situations where the attenuation of 186 keV U-235 gammas is expected to be greater than 90% is not recommended, due to the extreme sensitivity of this calculation to small errors in material thickness or density at high attenuation values.

Figure 2.4 is a graphical representation of the three source definitions available in HMS4. In the case of the point and line, the diameter of the disk or the width of the line cannot exceed 40% of the detector FOV.

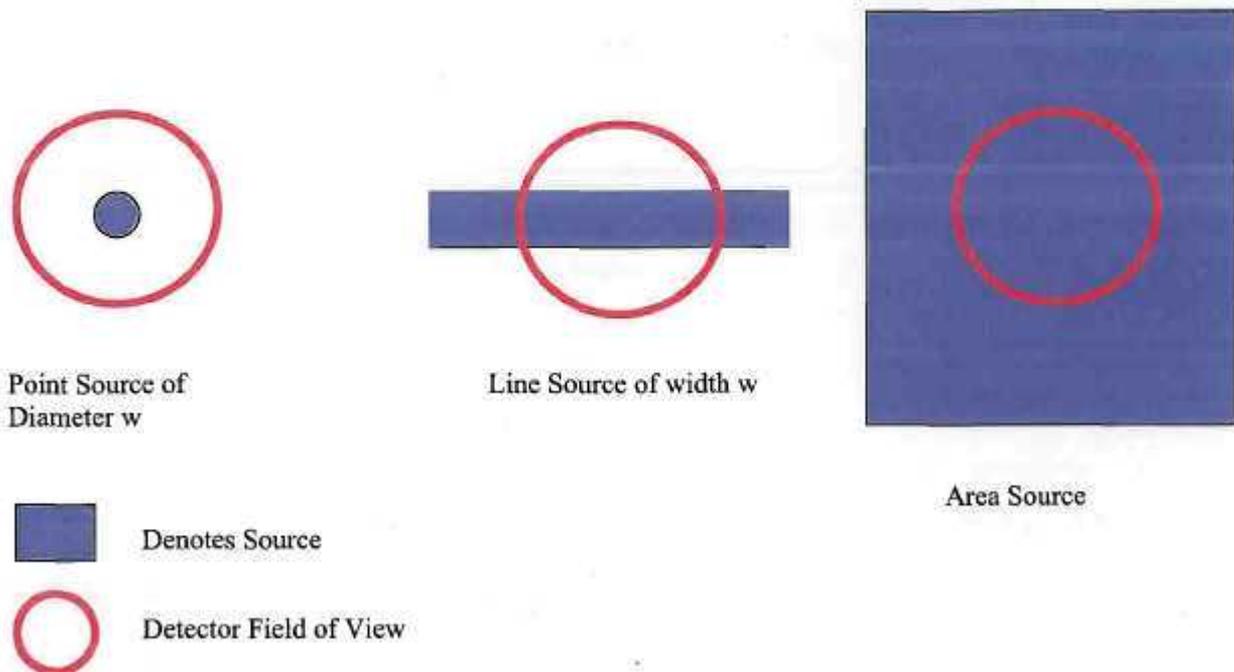


Fig. 2.4. HMS4 source type definitions.

HMS4 U-235 measurement becomes valid when the HMS4 measurement geometry, set by the Nondestructive Assay (NDA) engineer, complies with the measurement geometries set by Russo (Russo 2005) which is the calibration mode for that instrument. To this extent, care must be taken by the NDA engineer to ensure that the diameter of the point source or the width of the line source is small compared to the detector FOV. For an area source, the detector needs to be placed so that the edges of the deposit completely extend outside the FOV of the detector.

HMS4 performs calculations using the GGH algorithms developed at LANL (ORNL 2007). The GGH algorithms define the point, line, and area method of calculating the mass of U-235 contained in the sample. Calibration constants for point, line and area are derived during the calibration of the HMS4 system (Sect. 4.2 Calibration) and then U-235 mass calculations can be made for each generalized geometry:

$$\text{Point: U-235 grams} = K_{(p)} * CR * r^2 * CF_{sa} * CF_{fw} * CF_{ma} \quad (2.2)$$

$$\text{Line: U-235 grams} = K_{(l)} * CR * r * l * CF_{sa} * CF_{fw} * CF_{ma} \quad (2.3)$$

$$\text{Area: U-235 grams} = K_{(a)} * CR * a * CF_{sa} * CF_{ma} \quad (2.4)$$

where,

$K_{(p)}$, $K_{(l)}$ and $K_{(a)}$ are constants for point, line and area sources derived by calibration measurements (gm-sec/cm²)

CR = Count Rate of measurement for peak area (with background subtracted)

r = distance from detector face to source

l = associated length of deposit

a = associated area of deposit

CF_{sa} , CF_{fw} , CF_{ma} are the correction factors defined in Sect. 1.2.

Rudimentary GGH point, line, and area models assume that point and line deposits have no width. HMS4 incorporates a finite-source correction that uses a finite-source width parameter to compensate for deposit geometries that deviate from this assumption (Russo 2005).

$$CF_{Finite} = 2n * [1 + G(\omega/2)]^{-n} \quad (2.5)$$

where,

$n_{Point} = 2$, $n_{Line} = 1$, ω is the deposit width and G is the normalized Gaussian fit to the radial response determined in calibration.

HMS4 determines the deposit gamma ray self-attenuation by using the finite-source width parameter to calculate a deposit thickness for point and line deposits. For area deposits this width parameter is not needed, since the program calculates gram/cm². The defining equation for this finite source width correction is (Russo 2005):

$$(\rho x) = -(\ln(1 - \mu(\rho x)_{meas})) / \mu \quad (2.6)$$

where,

(ρx) is the true areal density of the deposit corrected for self-attenuation.

$(\rho x)_{meas}$ is the areal density of the deposit calculated without self-attenuation

μ is the mass attenuation coefficient of the deposit.

All of the parameters used by the HMS4 software GGH algorithms are explained in detail in the "Gamma-Ray Measurements of Holdup Plant-Wide: Application Guide for Portable, Generalized Approach" by P. Russo, developed at Los Alamos (Russo 2005).

Figure 2.5 shows a software screen available on the HMS4 controller where data for each measurement are entered.

Point Area/Equipment:	0101	Area Bkg Correction Flag:	0
Point Location:	001	Area Bkg Correction Factor:	0
Modified (User ID.):	025706	Gamma Energy (KeV):	105
Modified (Date):	12/10/03	Attenuating Material (E or C):	E
Modified (Time):	15:25	Atten. Material (Atomic Sym.):	FE
Source (P, L, or A):	P	Material Thickness (cm):	0.2
Measurement Distance (cm):	40	Material Density (g/cm ³):	0
Point Dia. or Line Width (cm):	0	μ for Material (cm ² /g):	0.14431018
Uncertainty in Pt/Line Width:	0	Equipment Wall Corr. Factor:	1.2546407
Associated Area (cm ²):	0	Finite Source Corr. Flag (ON/OFF):	OFF
Area Uncertainty (cm ²):	0	Self-Attenuation Corr. Factor:	1
Associated Length (cm):	0	Finite Source Corr. Factor:	1
Length Uncertainty (cm):	0	Extra Correction Factor:	1
Alarm Point (cnt/s):	2000	SIM Enrichment (%):	92
		SIM Material Type:	UO2

INVALID MEASUREMENT POINT - DO NOT USE!

Fig. 2.5. HMS4 controller input screen.

Using the interface, the NDA, engineer after evaluating the items to be in situ measured, inputs the parameters previously discussed to model those items in an appropriate manner which will render a correct calculation for the items. A simple description for each parameter is listed below:

Table 2.1 HMS4 input parameter definitions

Parameter	Description
Point Area/Equipment:	First 4 digits of 7 digit description of item being measured (Grouping number)
Point Location:	Last 3 digits of 7 digit description of item being measured (Specific Location)
Modified (User ID):	Badge # of NDA Engineer who last input parameters
Modified (Date):	Last Date modification of parameters
Modified (Time):	Time of last modification of parameters
Source (P, L or A):	Choice of Point, Line or Area geometry for source of item being measured
Measurement Distance(cm):	Distance of detector face to estimated location of source
Point Dia. Line Width(cm):	If Point or Line is chosen as source, the estimated width for finite source correction in cm is input. This information can sometimes be provided by process knowledge. If no process knowledge information is provided a conservative value such as the inner width of the pipe or container, or the largest width reasonably thought possible for the measurement is used. For Area this is not used.
Uncertainty in Pt/Line Width(cm):	Not used at the current time.
Associated Area (cm ²):	If an Area is chosen the Area of the source in cm ² is input. Normally the NDA engineer input for an area of inner diameter for 1 ft. of pipe. ($\pi d h$ or $\pi * \text{inner diameter} * 30.48\text{cm}$)

Table 2.1. HMS4 input parameter definitions (cont.)

Parameter	Description
Area Uncertainty (cm ²):	Not used at the current time.
Associated Length(cm):	If a Line is chosen the length * width of the source in cm ² is input. Normally we input for width of pipe and a length 1 ft. of pipe. (1*w or 30.48cm * width of pipe)
Length Uncertainty (cm):	Not used.
Alarm Point(cnt/s):	Triggers an alarm if cnt/s limit entered here is exceeded. Not used.
Area Bkg Correction Flag:	If an Area is chosen, the correction flag for bkg correction is input. Normally the bkg flag chosen is 2 which is source fills inner diameter of pipe. (See Fig. 2.3)
Area Bkg Correction Factor:	If an Area, based on the input above, HMS4 computes a bkg correction using calculations shown in Fig. 2.3
Gamma Energy (keV):	Gamma Energy in keV input here to determine attenuation corrections. Normally, 186 keV is input.
Attenuating Material (E or C):	Choice is made of Element or Compound of attenuating material.
Atten. Material (Atomic Sym):	Input is either one specific Element or Compound. If Element, HMS4 provides a density, if a Compound, density must be calculated or looked-up from common references and entered in Material Density parameter. Used in conjunction with "slab attenuation" dialog box to calculate values for attenuation.
Material Thickness(cm):	Thickness of attenuating material is input in cm. Normally this is the wall thickness of pipe. Process knowledge is used to input this value, but thickness gauges are employed to verify historical information and to provide a value when the information is not known. Used in conjunction with "slab attenuation" dialog box to calculate values for attenuation.
Material Density(g/cm ³):	Automatically enters if Element was chosen, or NDA Engineer hand inputs if Compound was chosen. . Used in conjunction with "slab attenuation" dialog box to calculate values for attenuation.
M for material (cm ² /g):	Generated by HMS4 based on the five previous parameters
Equipment Wall Corr. Factor:	Calculated by HMS4 based on the parameters thus far entered
Finite Source Corr. Flag(On/Off):	On is chosen if Finite Source Correction is used, Off if not used.
Self-Attenuation Corr. Factor:	Can be used to enter Self-Attenuation Correction Factor manually if already known or given by process knowledge. Normally not used.
Finite Source Corr. Factor:	Can be used to enter Finite Source Correction Factor manually if already known or given by process knowledge. Normally not used.
Extra Correction Factor:	Can be used to enter Extra Correction Factor manually. Normally used if additional attenuating material is encountered such as foam in pipe or pipe inside a duct. $\mu\rho\chi / 1 - e^{-\mu\rho\chi}$
SNM Enrichment (%):	% Enrichment is entered. Entered based on process knowledge.
SNM Material Type:	Material of source is entered from list provided by HMS4. Normally this is UO ₂ F ₂

2.4 THE DYNAMIC AREA MEASUREMENT METHOD

Field measurements of piping inside the K-25 and K-27 Bldgs. are characterized by high background values and poor counting statistics. Moreover, static area measurements of pipes do not bring the entire interior surface of the pipe into the detector FOV. To improve the foreground to background ratios and assure that the entire interior of a section of pipe is within the detector FOV, a "Dynamic Area Measurement Method" has been developed. In this technique, the detector is held with its axis always pointing through the axis of the pipe, and moved in serpentine fashion over one-half of the pipe such that the detector FOV intercepts 100% of the internal surface of the pipe over a specified period of time. Analysis of the reference technical document for the HMS4 system reveals that this technique is consistent with the development therein as long as the movement of the detector is smooth and comprehensive over the section of pipe. If significant nonuniformities exist in the deposited material, this technique assures that the nonuniform portion of the deposit (perhaps a significant buildup in a localized area) will be "seen" by the detector for some period of time.

To evaluate the capability of the "Dynamic Area Measurement Method," one can envision splitting a pipe into two half-pipes, each with a quadrature grid 15 strips wide as shown in Fig. 2.6.

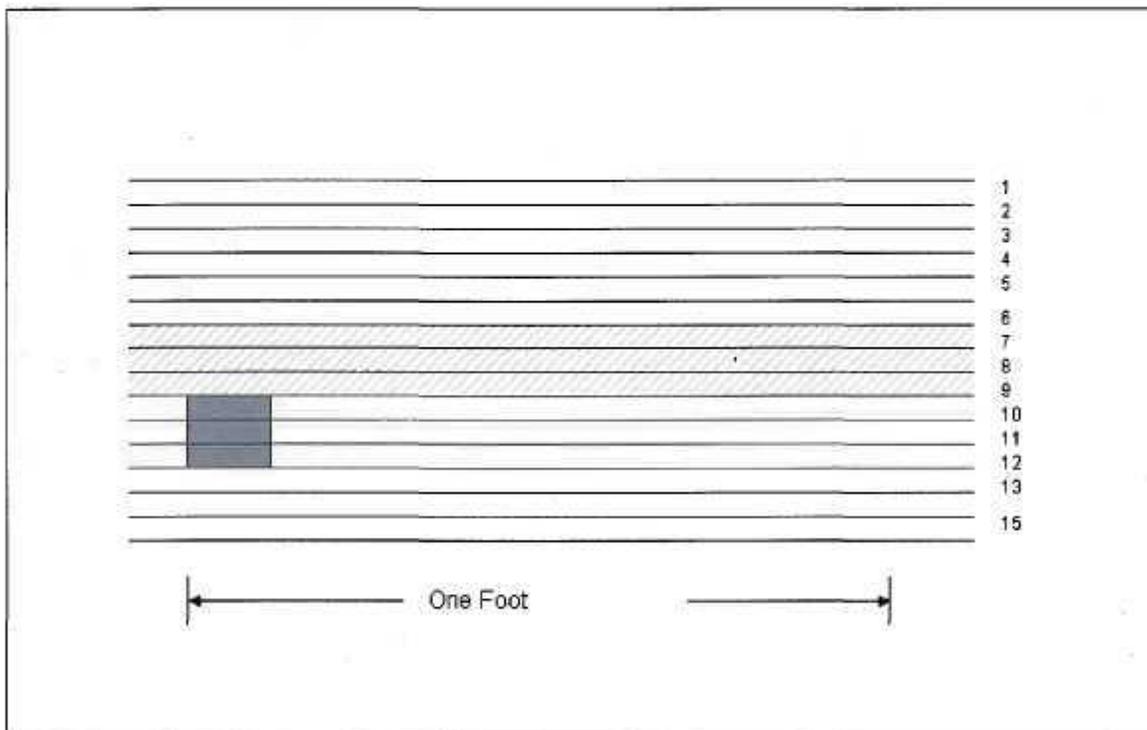


Fig. 2.6. Quadrature grid for HMS4 dynamic area measurement evaluation.

In this evaluation, visualize the two half-pipes as planes, one located above the other. For simplicity of math, assume the collimator is square and deep, so the FOV of the detector is the same for both planes, and is exactly three quadrature strips square.

For the case of uniform contamination over the entire interior of the pipe

$$GMS_a = \int_0^T R(t)C_R(t)dt \quad (2.7)$$

where,

GMS_a is the measured grams of U-235 using the area source algorithm.

$R(t)$ is the entire Russo (Russo 2005) area source data reduction algorithm including self-attenuation, matrix attenuation, source configuration (the detector has 2 source planes in its FOV at any point in time), and detector efficiency.

$C_R(t)$ is the net ROI 4 count rate at any point in time t

In a uniform pipe with uniform contamination,

$$R(t) = \text{constant} = R$$

$$C_R(t) = \text{constant} = C_R$$

$$GMS_a = \int_0^T RC_R dt = RC_R T \quad (2.8)$$

This is the same result that would be obtained from a static area source measurement.

If all the contamination in the pipe oxidizes, hydrates, and collapses into the bottom 3 quadrature strips of the pipe (this would be strips 7, 8, and 9 in Fig. 2.6), then

$$GMS_a = \int_0^T R(t)C_R(t)dt \quad (2.9)$$

and by numerical integration

$$GMS_a = N \left\{ \sum_{i=1}^5 R(t_i)C_{R_i}(t_i)\Delta t + \sum_{i=6}^{10} R(t_i)C_{R_i}(t_i)\Delta t + \sum_{i=11}^{15} R(t_i)C_{R_i}(t_i)\Delta t \right\} \quad (2.10)$$

where, N is the number of passes (lateral) required to traverse the entire 1 foot length of pipe.

and, $C_{R_i}(t_i) = 0$ for $i = 1-5$ and $i = 11-15$

$$\text{thus, } GMS_a = N \sum_{i=6}^{10} R(t_i)C_{R_i}(t_i)\Delta t \quad \text{where, } \Delta t = T/(15*N) \quad (2.11)$$

If self-attenuation is negligible, then $R(t) = R$ (a constant)

$$GMS_i = (RT/15) \sum_{i=6}^{10} C_R(t) \quad (2.12)$$

Define a_i as the fraction of $5 \cdot C_R$ that is counted in Δt_i when the detector FOV is centered over strip i .

(Note: The "5" comes from the fact that in the case of uniform contamination, the FOV is 6 quadrature strips worth of contamination, while if all the contamination is in the bottom 3 strips, the FOV if centered on strip 8 is seeing all 30 strips if contamination: $30/6 = 5$).

$$C_R(t_6) = CR(t_6) = a_6 \cdot 5 \cdot CR \quad (2.13)$$

$$C_R(t_7) = CR(t_7) = a_7 \cdot 5 \cdot CR \quad (2.14)$$

$$C_R(t_8) = 5 \cdot CR \quad (2.15)$$

$$GMS_i = ((5RTC_R)/15) \{2 \cdot a_6 + 2 \cdot a_7 + 1\}$$

To calculate the values of a_i , use the radial response curve in Fig. 4.2 approximated as a triangle of height 1 and base 17. The total area of the triangle is $(1/2) \cdot 17 \cdot 1 = 8.5$.

The fraction of the FOV that is in the detector FOV at position 6 is $A_6 = (1/2)(17/3)(2/3)/8.5 = 0.222$

and $a_7 = \{ (1/2)(17/3)(2/3) + (2/3)(17/3) - (1/2)(17/3)(1/3) \} / 8.5 = 0.778$

$$GMS_i = ((5RTC_R)/15) \{2 \cdot 0.222 + 2 \cdot 0.778 + 1\} = RC_R T \quad (2.16)$$

This is also the same result as the static measurement, and the same result as the dynamic measurement in the case of uniform contamination.

2.5 GAIN STABILIZATION AND IN-PROCESS CALIBRATION CHECKS

The HMS4 detector software is designed to provide quality control to ensure the data collected meets the standard necessary for the user. Two types of quality control tests are performed using the HMS4 software. The first test is performed after the completion of every acquisition. This test checks the Am-241 reference peak for gain shift and count rate. The count rate of the U-235 analysis peak is also confirmed with a check source at prescribed intervals.

The NDA engineer sets the number of measurements which can be made between source check measurements by the HMS4 software according to Bechtel Jacobs Company LLC (BJC) procedure BJC-DE-0708 (BJC 2007). Typically, this value is set to 11. This means that after 10 measurements of the background or foreground, a check source measurement would be required. The HMS4 software prompts the user to make quality control U-235 source checks initially and following each set of measurements. The software checks the counts per second of the U-235 peak against the information entered during setup and calibration to ensure the efficiency of the detector to detect U-235 is within the limits set by the user. The mean and two times the standard deviation are entered as the Check Source cps and the Check Source cps Limit, respectively. This real time quality check ensures the data collected meets the requirements necessary for the user and prompts the operator immediately when there is a problem.

If the quality control measurements fail but the detector can be adjusted, the measurements for that data set are remeasured to ensure the data meets the quality needed. If the detector can not be adjusted, the data set is rejected and will not be used. In addition the quality control check measurements are entered daily into a statistical program (MCCP) which compares the data collected with other data previously collected for that detector. Review of this data can establish trends and biases for further study to ensure the detector is functioning properly.

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3. MEASUREMENT CRITERIA

3.1 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) have been established by various users of NDA information. The tables below summarize the most restrictive DQOs for items that may be measured by the HMS4 system. In Table 3.1, DQOs are tabulated which HMS4 may be able to achieve under favorable (low) background conditions. These are referred to as "Provisional DQOs," meaning that they can be met in some field measurement situations, but not in others, where background levels are not optimum.

Table 3.2 provides DQOs and other data for large-bore pipe, and Table 3.3 provides this information for valves and expansion joints.

The "Multi-Agency Radiation Survey and Site Investigation Manual" (MARSSIM 1998) recommends that the Minimum Detectable Amount (MDA) in field radiation measurements be no more than 50% of a stated DQO. Use of HMS4 should follow this guidance wherever possible.

Table 3.1. Provisional DQOs for small-bore pipe and tubing

Item	Nominal (in.)	Material of Construction	OD (in.)	ID (in.)	Wall Thickness (in.)	Length (in.)	Area (cm ² /ft)	DQO U-235 (g/ft)
Tubing	0.25	Steel	0.540	0.364	0.088	12	89	0.5
	0.5	Steel	0.840	0.622	0.109	12	151	0.5
	0.75	Monel	1.050	0.824	0.113	12	200	0.5
	0.75	Steel	1.050	0.824	0.113	12	200	0.5
Copper Tubing	0.25	Copper	0.540	0.364	0.088	12	89	1
	0.5	Copper	0.840	0.622	0.109	12	151	1
	0.75	Copper	1.050	0.824	0.113	12	200	1
1-in. \leq diameter < 2-in.	1	Copper	1.315	1.049	0.133	12	255	1.5
	1	Monel	1.315	1.049	0.133	12	255	1.5
	1	Steel	1.049	1.000	0.025	12	243	1.5
	1.5	Copper	1.900	1.610	0.145	12	392	1.5
	1.5	Monel	1.900	1.610	0.145	12	392	1.5
	1.5	Steel	1.610	1.315	0.148	12	320	1.5
2-in. \leq diameter < 3-in.	2	Copper	2.375	2.067	0.154	12	503	3
	2	Monel	2.375	2.067	0.154	12	503	3
	2	Steel	2.375	2.067	0.154	12	503	3
	2.5	Steel	2.875	2.469	0.203	12	601	3

Table 3.2. DQOs for large-bore piping

Item	Nominal (in.)	Material of Construction	OD (in.)	ID (in.)	Wall Thickness (in.)	Length (in.)	Area (cm ² /ft)	DQO U-235 (g/ft)
3-in. ≤ diameter < 4-in.	3	Copper	3.500	3.370	0.065	12	820	7
	3	Monel	3.500	3.370	0.065	12	820	7
	3	Steel	3.500	3.068	0.216	12	746	7
	3.5	Steel	4.000	3.548	0.226	12	863	7
4-in. ≤ diameter < 6-in.	4	Copper	4.500	4.370	0.065	12	1063	10
	4	Monel	4.500	4.370	0.065	12	1063	10
	4	Steel	4.500	4.026	0.237	12	979	10
6-in. ≤ diameter < 10-in.	5	Steel	5.563	5.047	0.258	12	1228	10
	6	Steel	6.625	6.065	0.280	12	1475	14
	8	Steel	8.625	8.071	0.277	12	1963	14
diameter > 10-in.	10	Steel	10.750	10.192	0.279	12	2479	22
	12	Steel	12.750	12.090	0.330	12	2941	22
	14	Steel	14.000	13.375	0.313	12	3253	22
	16	Steel	16.000	15.375	0.313	12	3740	22

Table 3.3. DQOs for expansion joints and valves

Item	Nominal (in.)	Material of Construction	OD (in.)	ID (in.)	Wall Thickness (in.)	Length (in.)	Area (cm ² /ft)	DQO U-235 (g/ft)
Expansion Joints	3	Monel	3.500	3.370	0.065	12	889	10
	4	Monel	4.500	3.370	0.065	12	1021	10
	5	Monel	5.563	3.068	0.216	12	1161	10
	6	Monel	6.625	3.548	0.226	12	1301	10
	8	Monel	8.625	N/A	N/A	N/A	1564	10
	10	Monel	10.750	N/A	N/A	N/A	1844	10
	12	Monel	12.750	N/A	N/A	N/A	2108	10
	14	Monel	14.000	N/A	N/A	N/A	2273	10
	16	Monel	16.000	N/A	N/A	N/A	2536	10
Valves	3	Steel	6.875	6.000	0.4375	15.4225	1876	40
	4	Steel	6.875	6.000	0.4375	14.9225	1815	50
	6	Steel	9.6875	8.375	0.65625	21.03	3570	50
	8	Steel	12.000	10.750	0.625	16.3725	3567	50
	10	Steel	13.875	12.750	0.5625	23.075	5963	75
	12	Steel	16.250	15.000	0.625	32.75	9957	75
	14	Steel	17.500	16.000	0.75	36.5	11837	75
16	Steel	20.250	18.380	0.9375	42.75	15921	75	

3.2 PRECISION AND ACCURACY

Precision is a measure of the reproducibility of the system and is determined through replicate counting of an item with a known quantity of radioactive material of interest. For the purposes of this test program, precision is expressed as the % Relative Standard Deviation (RSD) for a set of replicate measurements and is calculated as follows:

$$\%RSD = (\sigma / \bar{x}) * 100 \quad (3.1)$$

where,

σ is the standard deviation,

\bar{x} is the mean of the replicate measured U-235 values.

Accuracy, or bias, is the degree of agreement between measured concentration or activity values and the true or known values. Accuracy is determined by replicate counting of containers with standards of known U-235 content. For this test program, accuracy is expressed as percent recovery, %R, and is calculated as follows:

$$\%R = (C_m / C_{sm}) * 100 \quad (3.2)$$

where,

C_m is the average of the replicate measured U-235 values.

C_{sm} is the "true" or certified value.

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4. INSTRUMENT QUALIFICATION

All calibration, calibration verification, and calibration confirmation measurements are to be executed utilizing current and applicable BJC procedures, and subcontractor procedures and work instructions.

4.1 INSTRUMENT SETUP

The following table defines instrument setup:

Table 4.1. Instrument setup test requirements

Performance Test	Performance Method	Test Results
Prepare instrument for routine operation by creating or developing requisite software and hardware parameters.	<p>Identify the NaI(Tl) detector MCA set for use.</p> <p>Identify and use a performance check source of U-235:</p> <ol style="list-style-type: none">1. Place the identified performance check source on or near (specify in results) the HMS4 detector.2. Establish and record optimum operational (setup) parameters for the instrument hardware and software. (See Fig. 4.1)3. Establish and record measurement control limit for acceptable performance based on measurements of the performance check source.4. Perform a background measurement before introducing the calibration source into the detector environment.	

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Figure 4.1 shows a typical energy spectrum obtained where a successful setup is obtained using a U-235 check source. The full energy peak from 186 keV is observed together with the full energy peak from the Am-241 60 keV check source installed as part of the HMS4. Regions of interest are also identified that are used to assess and subtract background events.

Typical Regions of Interest (ROI) for ²³⁵U

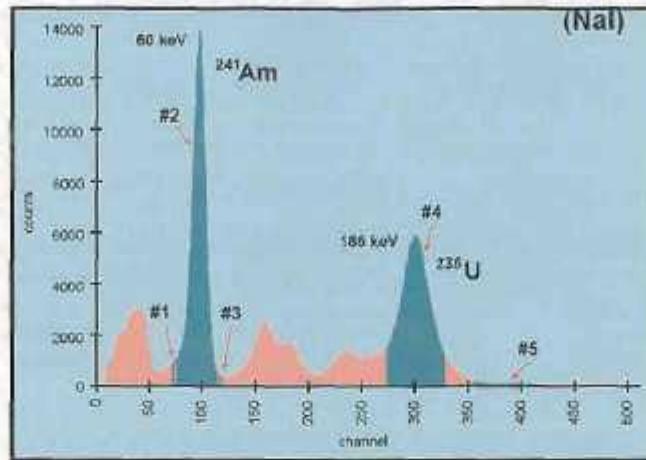


Fig. 4.1. Typical spectrum with assigned ROIs obtained after successful setup.

4.2 CALIBRATION

DOE M 470.4-6 (DOE 2005) requires that all measurement methods must be calibrated using (1) standard or certified reference materials, or (2) secondary standards traceable to the national measurement base, and must be revalidated as necessary. This protocol is shown in Table 4.2 below.

Table 4.2. Calibration test requirements

Performance Test	Performance Method	Test Results
<p>χ-squared Test to confirm that the instrument is counting random events, rather than noise or coherent signals.</p>	<p>Confirm operational readiness of the detector/MCA set.</p> <p>Identify and use a certified (NIST/NBL traceable) measurement standard source.</p> <p>Perform Measurements for Chi-Squared Test:</p> <ol style="list-style-type: none"> Utilizing the calibration fixture, place an approximately 10 g U-235 standard in a fixed position, horizontally centered on the detector axis, and 20-40 cm from the NaI(Tl) crystal front face. Perform six measurements of the source. Do not touch or disturb the source or detector between these measurements. The counting time is arbitrary, except that there should be about 400 counts in the peak ROI 4 for each measurement. Background subtraction should not be performed. Using these six measurements, perform the chi-squared test described in Sect. 4.2.1. 	

Table 4.2. Calibration test requirements (cont.)

Performance Test	Performance Method	Test Results
<p>Determine source positioning error contribution to Total Measurement Uncertainty</p>	<p>Perform R_0 Measurements</p> <ol style="list-style-type: none"> 1 Utilizing the calibration fixture place the 10 g U-235 standard in a fixed position, horizontally centered on the detector and 40 cm from the HMS4 detector face <p>(Note: The source - detector distance is from the detector NaI(Tl) crystal front face, not the detector housing)</p> <ol style="list-style-type: none"> 2 Perform six replicate measurements of the standard source. Ensure a new background is obtained for each replicate and that the source-detector setup positioning is performed for each replicate 3 Tabulate the measurement data for entry into the HMS4 calibration software 	
<p>Perform radial response measurements to determine point, line and area calibration constants</p> <p>Test to confirm undamaged crystal and proper collimator alignment</p>	<p>Perform Radial Response Measurements</p> <ol style="list-style-type: none"> 1 Utilizing the calibration fixture, place the 10 g U-235 standard in a fixed position, horizontally centered on the detector axis and 40 cm from the NaI(Tl) crystal front face 2 Repeat R_0 measurement 3 Perform nine successive measurements by moving the standard, in 5 cm increments, off the centerline to the right and then nine measurements to the left of the detector 4 Tabulate the measurement data for entry into the HMS4 calibration software 5 Rotate the detector 90° and repeat the Radial Response Measurements 6 Tabulate the measurement data for entry into the HMS4 calibration software 	
	<p>Perform initial calibration</p> <ol style="list-style-type: none"> 1 Enter the tabulated data collected above using HMS4 2 Print HMS4 MCA/Detector calibration report <p>Perform rotated calibration</p> <ol style="list-style-type: none"> 1 Enter the tabulated data collected above using HMS4 2 Print HMS4 MCA/Detector calibration report 	

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NIST National Institute of Standards and Technology

NBL New Brunswick Laboratory

Table 4.3. Calibration results

Chi-Squared Test (per equation [4.1])

Instrument Identification: _____

First Test _____

Second Test (If necessary) _____

Third Test (If necessary) _____

Replicate Measurements at $R_c = 40$ cm (Net Counts, ROI 4 (per equation [2.1]))

Measurement	Source + Background		Background		Net Counts
	ROI 4	ROI 5	ROI 4	ROI 5	ROI 4
1					
2					
3					
4					
5					
6					

Source Positioning Variance

$\sigma_{sp}^2 =$ _____ (Per equation [4-7])

%RSD= _____ (Per equation [4-9])

Radial Response Measurements (Net Counts, ROI 4 per equation [2-1])

	Centimeters Offset																			
	45	40	35	30	25	20	15	10	5	0	5	10	15	20	25	30	35	40	45	
Base																				
90° Rotated																				

Calibration Constants (as reported by HMS4)

Point Source (K_p) _____ Line Source (K_l) _____ Area Source (K_a) _____

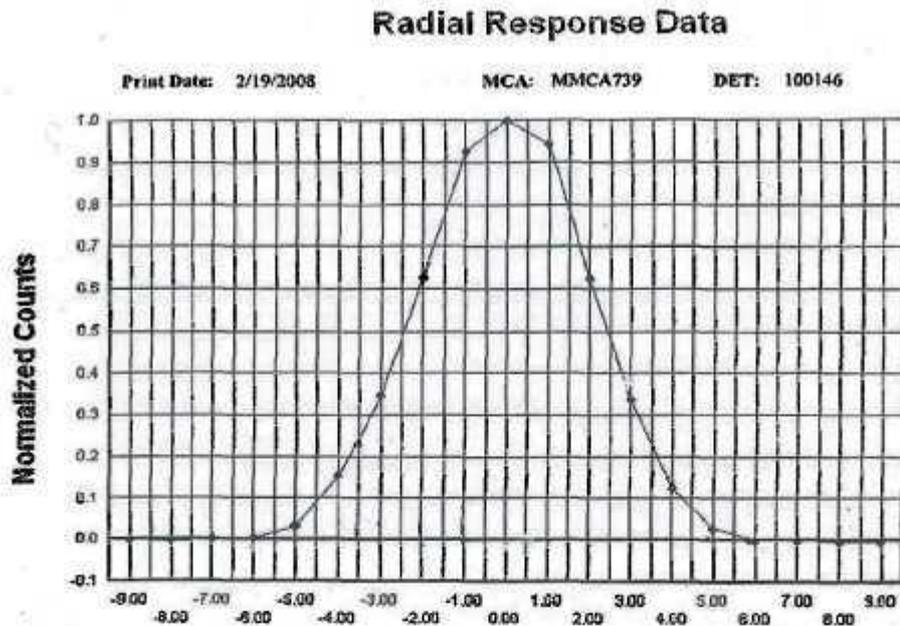


Fig. 4.2. Typical response curve after completion of calibration.

All these data may be viewed through the HMS4 calibration interface window as shown in Fig. 4.3.

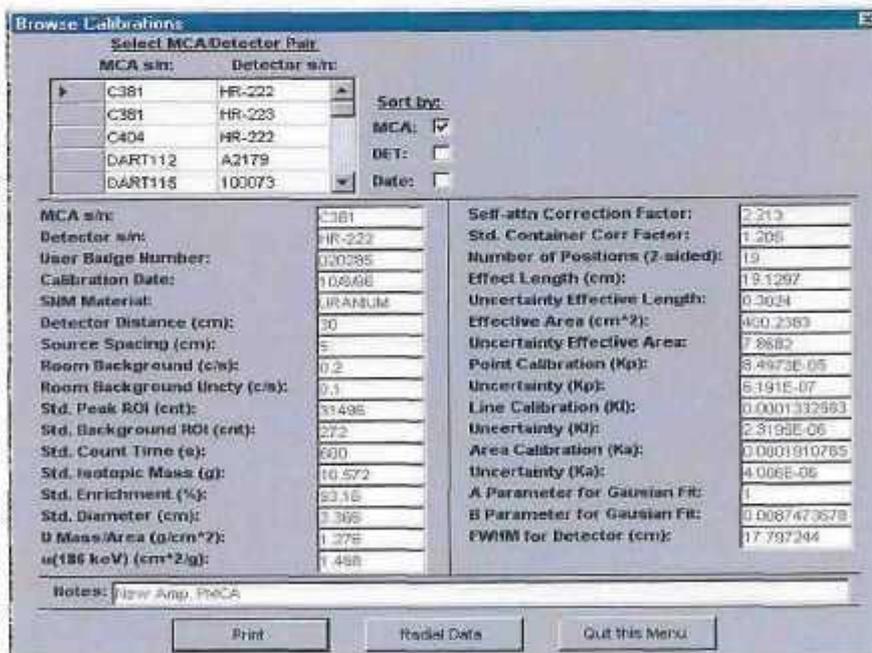


Fig. 4.3. HMS4 calibration interface.

The above calibration process follows the guidance contained in the base reference document for the HMS4 system (Russo 2005). An additional step has been added—“Rotate the detector 90° and repeat the radial response measurements.” This step is designed to detect radial source positioning errors, and nonuniform response of the detector/collimator assembly. Application of this additional data is discussed in Sect. 5.4.2.1.4, Other Potential Calibration Errors.

4.2.1 Chi-Squared Test

To confirm that the detector is operating in response to random events (rather than white noise or bleed-over from system power or electronics), a chi-squared test will be run on six replicate measurements with the source on the axis of the detector 40 cm from the face of the detector. The source-geometry configuration will not be touched or disturbed in any way between these six measurements. These measurements will be performed before the calibration is initiated.

The chi-squared test equation is:

$$\chi^2 = (1/x_{\text{mean}}) \sum_{i=1}^6 (x_i - x_{\text{mean}})^2 \quad (4.1)$$

where, x_{mean} is the mean counts (no background subtraction) of the six raw chi-squared test measurements.

x_i are the counts for the individual raw chi-squared test measurements.

Acceptable values of χ^2 for six measurements range from 1.610 to 9.236. Outside this range, there is more than an 80% chance that extraneous non-random events are being counted. If the value is outside this range, the problem must be diagnosed and rectified before proceeding to reperform the chi-squared test. The first step in the diagnosis should be to reperform the chi-square test twice. If both tests are within the acceptable limits, the first test was probably a statistical outlier. If one or both fail, proceed to diagnose the problem.

4.2.2 Calculation of Source Positioning Error Contribution to TML

The six replicate calibration measurements in Table 4.2 of the source on the axis of the detector at a distance of 40 cm from the face of the detector (under "Perform R_0 Measurements") also provides for the calculation of the contribution to the total measurement uncertainty of source positioning error in calibration, since the source-detector geometry is reconstructed after each measurement.

If, as is believed, the variance in the replicate measurements is composed of counting errors and source positioning errors, then the variance in the replicate measurements is

$$\sigma_{\text{repl}}^2 = \sigma_{\text{counting}}^2 + \sigma_{\text{sp}}^2 \quad (4.2)$$

where, σ_{repl}^2 is the variance of the net replicate counts in the ROI.

$\sigma_{\text{counting}}^2$ is the variance of the net replicate counts due to counting statistics.

σ_{sp}^2 is the variance of the net replicate counts due to source position.

Rearranging gives:

$$\sigma_{sp}^2 = \sigma_{ROI}^2 - \sigma_{counting}^2 \quad (4.3)$$

The net counts in ROI 4 for a single measurement are:

$$x_{ROI4} = x_{i4} - (x_{b4} - x_{b5}) - x_{i5} \quad (4.4)$$

The mean value of the replicate measurements is:

$$x_{ROI4,mean} = \sum_{i=1}^N (x_{i4} - x_{i5} - x_{b4} + x_{b5})/N \quad (4.5)$$

where, N is the number of measurements (6)

x_{ij} is the measurement number i in ROI j .

The mean of any of the ROI measurements is

$$x_{j,mean} = \sum_{i=1}^N x_{ij}/N \quad (4.6)$$

Accounting for the data reduction to subtract background, and assuming that the variance of the count rates in any ROI is approximated by the mean counts in that region gives:

$$\sigma_{sp}^2 = \left\{ \sum_{i=1}^N (x_{i4} - x_{i5} - x_{b4} + x_{b5} - x_{ROI4,mean})^2 / (N-1) - \{x_{4,mean} - x_{5,mean} - x_{b4,mean} + x_{b5,mean}\} \right\} \quad (4.7)$$

where, $x_{b4,mean}$ - $x_{b5,mean}$ are the background values in ROI 4 and 5.

also, $\sigma_{sp} = (\sigma_{sp}^2)^{1/2}$ (counts) (4.8)

which gives the percent relative standard deviation due to calibration source positioning as

$$\%RSD_{sp} = 100 \sigma_{sp}/x_{ROI4,mean} \quad (4.9)$$

and, the effect of calibration source positioning error on field measurements of U-235 mass is

$$\sigma_{sp} \text{ (grams U-235)} = \%RSD_{sp} (m_{meas})/100 \quad (4.10)$$

where, m_{meas} is the measured point, line, or area source grams U-235 in an HMS4 field measurement.

σ_{sp} (grams U-235) is the parameter used in Equation (5.3) to compute total measurement uncertainty.

4.3 CALIBRATION VERIFICATION

As previously stated, valid holdup assessment with the HMS4 in field deployment demands that the HMS4 usage complies with the calibration geometries. Verification of calibration geometries is therefore obviously critical.

Modeling of the equipment is based on how the item is positioned relative to the detector's FOV. The methods to be validated are for a point, line, or area source. The following describes the item positioning required for each method.

- A point source simulates a small, localized deposit that is completely inside the detector's FOV. A point source should occupy 40% or less of the diameter of the detector FOV.
- A line (simulating small pipe deposition) source is a narrow source that extends beyond the detector's FOV in one dimension. A line source fills the FOV from one side to the other while occupying 40% or less in the other dimension.
- An area source (simulating large duct, pipe, floor, etc.) extends beyond the detector's FOV in all directions.

Table 4.4 prescribes calibration verification test requirements. Results of these tests are to be reported as defined in Table 4.5.

Table 4.4. Calibration verification requirements

Performance Test	Performance Method	Test Results
<p>Point source verification</p> <p>Second point source measurement to confirm linearity</p>	<p>Confirm operational readiness of the detector MCA part</p> <p>Identify and use qualified measurement standard(s)</p> <p>Perform 'point source' verification</p> <ol style="list-style-type: none"> 1 Utilizing the calibration fixture or appropriate detector holders or hardware, place a U-235 "point source" standard (at least a secondary [well-characterized] standard) of between 1 and 5 grams U-235 in a fixed position, horizontally centered on the detector, and at a distance less than 15 cm from the detector face which conforms to the requirements for a point source <p>Note: The detector distance is from the detector itself, not the detector housing</p> <ol style="list-style-type: none"> 2 Perform three replicate measurements of the standard 3 Tabulate the measurement data 4 Use the appropriate HMS4 "point source" calibration constants to determine the U-235 mass of the measured standard 5 Calculate and report the measured accuracy and precision for the "point source" verification measurement 6 Repeat steps 1-5 using a well-characterized source of approximately 10 grams U-235 	
<p>Line source verification</p>	<p>Perform "line source" verification</p> <ol style="list-style-type: none"> 1 Utilizing the calibration fixture or appropriate detector holders or hardware, place a U-235 "line source" standard (at least a secondary [well-characterized] standard) in a fixed position horizontally centered on the detector from the detector face at a distance which conforms to the requirements of a line source 2 Perform three replicate measurements of the standard 3 Tabulate the measurement data 4 Use the appropriate HMS4 "line source" calibration constants to determine the U-235 mass of the measured standard 5 Calculate and report the measured accuracy and precision for the "line source" verification measurement 	

Table 4.4. Calibration verification requirements (cont.)

Performance Test	Performance Method	Test Results
Area source verification.	<p>Perform "area source" verification:</p> <ol style="list-style-type: none"> 1 Utilizing the calibration fixture or appropriate detector holders or hardware, place a U-235 "area source" standard (at least a secondary [well-characterized] standard) in a fixed position horizontally centered on the detector at a distance which conforms to the requirements of an area source 2 Perform three replicate measurements of the standard 3 Tabulate the measurement data 4 Use the appropriate HMS4 "area source" calibration constants to determine the U-235 mass of the measured standard. 5 Calculate and report the measured accuracy and precision for the "area source" verification measurement 	

MCA Multi-channel analyzer

In the point, line, and area source tests described in Table 4.4, three replicate measurements are made for each source type. The accuracy of each is defined by the following equations:

$$m_{\text{mean}} = (m_1 + m_2 + m_3) / 3 \quad (4.11)$$

where,

m_{mean} is the mean measured U-235 mass in grams.

m_1, m_2, m_3 are the U-235 mass results from the three measurements.

The percent recovery of the measurements is defined as:

$$\%R = 100 (m_{\text{mean}}) / m \quad (4.12)$$

where,

m is the known mass of the calibration standard.

The precision for each three-replicate test is defined by the relative standard deviation as

$$\%RSD = 100 \left[\left\{ (m_{\text{mean}} - m_1)^2 + (m_{\text{mean}} - m_2)^2 + (m_{\text{mean}} - m_3)^2 \right\} / 2 \right]^{1/2} / m_{\text{mean}} \quad (4.13)$$

The success criterion for this test is based on historic evaluations of the performance of this type of instrument (NRC 1991a): These evaluations anticipate a total measurement uncertainty of 50% for field measurements with attenuating materials. Performance data for this instrument are not available, but for measurement of sources in air, the performance should be markedly superior to measurements in attenuating materials. Thus, the initial success will be:

$$100 - \%R < 25\% \quad (4.14)$$

If this inequality is violated, the test should be reperfomed at least twice (six additional measurements) and the %R and %RSD recomputed on the basis of the available nine measurements. If the inequality is still violated, it is very likely that a systematic error of some nature is present in the calibration or calibration verification measurements and/or data reduction. In this case, the causal factor(s) must be diagnosed and remedied, and the calibration and/or calibration verification measurements reperfomed.

Table 4.5. Calibration verification results

Instrument Identification: _____

Point Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [4.11]) %R _____ % (equation [4.12]) %RSD _____ % (equation [4.13])		
Line Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [4.11]) %R _____ % (equation [4.12]) %RSD _____ % (equation [4.13])		
Area Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [4.11]) %R _____ % (equation [4.12]) %RSD _____ % (equation [4.13])		
Second Point Source (Known Source _____ grams U-235)		
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [4.11]) %R _____ % (equation [4.12]) %RSD _____ % (equation [4.13])		

5. METHOD QUALIFICATION

5.1 CALIBRATION CONFIRMATION

The fundamental qualification of the HMS4 system is to demonstrate the ability of the instrument and its associated analysis algorithms to report a measured value of U-235 within the value given for a known sample. The tests described in Table 5.1 will be conducted to determine the consistent repeatability (precision) of the detector with items similar to those for which the detector will be used. The instrument accuracy reflects the degree to which the measured quantity can be algorithmically treated to render a calculated value that matches a known value. Both accuracy and precision characteristics, given the measuring instrument and standard methodology, are essential to establish the abilities and limitations of the instrument to perform a certain task.

Table 5.1. Calibration confirmation test requirements

Performance Test	Performance Method	Test Results				
<p>Demonstrate the capability of the HMS4 calibration algorithms to correctly report known material content in surrogates that are representative of actual material holdup configurations.</p> <p>Static point source</p>	<p>Confirm operational readiness of the detector/MCA pair. Identify and use qualified measurement standard(s).</p> <p>Perform "point source" confirmation:</p> <table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th style="text-align: center;">Type</th> <th style="text-align: center;">Configuration</th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">Pipe</td> <td style="text-align: center;">Carbon steel, schedule 40 ~ 3-in. diameter</td> </tr> </tbody> </table> <ol style="list-style-type: none"> 1. Place a U-235 "point source" standard (at least a secondary (well-characterized) standard) in a material matrix that is representative of material configurations for HMS4 measurements. 2. Model and measure the point source using FSG-WI-13. 3. Perform three replicate measurements of each of the surrogate configurations. 4. Tabulate the measurement data as shown in Table 5.2. 	Type	Configuration	Pipe	Carbon steel, schedule 40 ~ 3-in. diameter	
Type	Configuration					
Pipe	Carbon steel, schedule 40 ~ 3-in. diameter					

Table 5.1 Calibration confirmation test requirements (cont.)

Performance Test	Performance Method	Test Results						
<p>Static line source</p>	<p>Perform "line source" confirmation</p> <ol style="list-style-type: none"> 1 Place a U-235 "line source" standard (at least a secondary [well-characterized] standard) in a material matrix that is representative of material configurations for HMS4 measurements * <table border="1" data-bbox="624 506 1144 659"> <thead> <tr> <th>Type</th> <th>Configuration</th> </tr> </thead> <tbody> <tr> <td>Pipe</td> <td>Carbon steel, schedule 40 < 3-in diameter</td> </tr> <tr> <td>Pipe</td> <td>Carbon steel, schedule 40 > 6-in diameter</td> </tr> </tbody> </table> <ol style="list-style-type: none"> 2 Model and measure the "line source" using FSG-WI-13 3 Perform three replicate measurements of each of the surrogate configurations 4 Tabulate the measurement data as shown in Table 5.2 	Type	Configuration	Pipe	Carbon steel, schedule 40 < 3-in diameter	Pipe	Carbon steel, schedule 40 > 6-in diameter	
Type	Configuration							
Pipe	Carbon steel, schedule 40 < 3-in diameter							
Pipe	Carbon steel, schedule 40 > 6-in diameter							
<p>Static area source</p>	<p>Perform static "area source" confirmation</p> <ol style="list-style-type: none"> 1 Place a U-235 "area source" standard (at least a secondary [well-characterized] standard) in a material matrix that is representative of material configurations for HMS4 measurements <table border="1" data-bbox="624 1106 1144 1202"> <thead> <tr> <th>Type</th> <th>Configuration</th> </tr> </thead> <tbody> <tr> <td>Pipe</td> <td>Carbon steel, schedule 40 > 4-in diameter</td> </tr> </tbody> </table> <ol style="list-style-type: none"> 2 Model and measure the "area source" using FSG-WI-13 3 Perform three replicate measurements of each of the surrogate configurations 4 Tabulate the measurement data as shown in Table 5.2 	Type	Configuration	Pipe	Carbon steel, schedule 40 > 4-in diameter			
Type	Configuration							
Pipe	Carbon steel, schedule 40 > 4-in diameter							
<p>Dynamic area source</p>	<p>Perform "dynamic area source" confirmation</p> <ol style="list-style-type: none"> 1 Without disturbing the static area source confirmation source/material matrix configuration, apply the dynamic area source measurement technique to this configuration 2 Perform three replicate measurements of this configuration 3 Tabulate the measurement data as shown in Table 5.2 							

FSG - Fairfield Service Group

* Since piping measurements are usually carried out in one-foot sections at K-25 K-27, the point, line, and area source standards should be placed in a known one-foot section of the surrogate pipe.

In the point, line, and area source tests described in Table 5.1, three replicate measurements are made for each source type. The accuracy of each is defined by the following equations:

$$m_{mean} = (m_1 + m_2 + m_3)/3 \quad (5.1)$$

where,

m_{mean} is the mean measured U-235 mass in grams.

m_1 , m_2 , m_3 are the U-235 mass results from the three measurements.

The percent recovery of the measurements is defined as

$$\%R = 100 (m_{mean})/m \quad (5.2)$$

where, m is the known mass of the calibration standard.

The precision for each three-replicate test is defined by the relative standard deviation as

$$\%RSD = 100 \left[\left\{ (m_{mean} - m_1)^2 + (m_{mean} - m_2)^2 + (m_{mean} - m_3)^2 / 2 \right\}^{1/2} \right] / m_{mean} \quad (5.3)$$

The success criterion for this test is based on historic evaluations of the performance of this type of instrument (NRC 1991a), these evaluations anticipate a total measurement uncertainty of 50% for field measurements with attenuating materials. Since performance data for this instrument type are not available, the initial success criterion will be

$$100 - \%R_1 < 50\% \quad (5.4)$$

If this inequality is violated, the test should be reperfomed at least twice (six additional measurements) and the %R and %RSD recomputed on the basis of the available nine measurements. If the inequality is still violated, it is very likely that a systematic error of some nature is present in the calibration or calibration verification measurements and/or data reduction. In this case, the causal factor(s) must be diagnosed and remedied, and the calibration and/or calibration verification measurements reperfomed.

Table 5.2. Calibration confirmation results

Instrument Identification: _____

Point Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.2])		
%RSD _____ % (equation [5.3])		
Line Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.2])		
%RSD _____ % (equation [5.3])		
Area Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.2])		
%RSD _____ % (equation [5.3])		
Dynamic Area Source (Known Source _____ grams U-235)		
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.2])		
%RSD _____ % (equation [5.3])		

5.2 MODEL VARIABILITY

The purpose of the measurements in Table 5.3 is to determine the extent to which a point or line deposit can be detected and measured using the static or dynamic area source measurement algorithm in HMS4 routine field measurements.

The K-25 vent, purge, and drain (VPD) program will have visually inspected piping and components for deposits. Of concern in piping measurements is the potential that oxidation and hydration of UF_6 deposits to $UO_2 \cdot xH_2O$ may have caused accumulation of material into a "point" or "line" source in the bottom of the pipe after the visual inspection. In general, pipe with observed deposits will have been removed from the building, but post-inspection accumulation could have occurred.

The fundamental goal in such situations is that the deposit is detected and quantified in some fashion. The specified measurements will be utilized to define a "percent recovery" (accuracy) for area source measurements of point and line sources from the top, side, and bottom of piping systems.

Table 5.3. Model variability test requirements

Performance Test	Performance Method	Test Results
<p>Determine the variability of the reported U-235 mass values when the area algorithm is used to measure sample configurations that actually display point or line characteristics</p>	<p>Confirm operational readiness of the detector/MC A pair</p> <p>Identify and use qualified measurement standard(s)</p> <p>Perform "point" source evaluation</p> <ol style="list-style-type: none"> 1 Position the HMS4 detector to emulate standard holdup measurement protocols 2 Place a well characterized point source standard in a surrogate, carbon steel, 3-in. diameter pipe * Model the measurements as an area source by following FSG-WI-13 3 Perform three replicate measurements applying the dynamic measurement technique from the top of the pipe 4 Perform three replicate measurements applying the dynamic measurement technique from the side of the pipe 5 Perform three replicate measurements applying the dynamic measurement technique from the bottom of the pipe 6 Calculate and report the measured accuracy and precision for each set of three replicate measurements as shown in Table 5.4 	

Table 5.3. Model variability test requirements (cont.)

Performance Test	Performance Method	Test Results
	<p>Perform "line" source evaluation</p> <ol style="list-style-type: none"> 1 Position the HMS4 detector to emulate standard holdup measurement protocols 2 Place a well characterized line source standard in a surrogate, carbon steel, >4-m diameter pipe * Model the measurements as an area source by following FSG-WI-13 3 Perform three replicate measurements applying the dynamic measurement technique from the top of the pipe 4 Perform three replicate measurements applying the dynamic measurement technique from the side of the pipe 5 Perform three replicate measurements applying the dynamic measurement technique from the bottom of the pipe 6 Calculate and report the measured accuracy and precision for each set of three replicate measurements as shown in Table 5.4 	

* Since piping measurements are usually carried out in one-foot sections at K 25 K 27, the point, line, and area source standards should be placed in a known one-foot section of the surrogate pipe.

The percent recovery of a point source measured as an area source is (for three replicate measurements)

$$\%R_{pa} = (100/3) m_{pt} \sum_{i=1}^3 m_{meas,pt} \quad (5.5)$$

where, $m_{meas,pt}$ are the individual measured mass values of the point source and

m_{pt} is the true (known) mass of the point source

The percent recovery of a line source measured as an area source is

$$\%R_{la} = (100/3) m_l \sum_{i=1}^3 m_{meas,l} \quad (5.6)$$

where, $m_{meas,l}$ are the individual measured mass values of the line source

m_l is the true (known) mass of the line source.

In piping systems, due to gravity, it is probable that undetected point or line source deposits will fall to the bottom of the pipe. If this is the case, area source algorithm measurements made from the side of the pipe may not detect some or all of the deposits in the bottom of the pipe. This geometric configuration will also cause under-prediction of the attenuation of 186 keV gamma rays from U-235, which will cause a negative bias to the U-235 mass measurement. (The track length of a gamma ray coming from a deposit in the bottom of the pipe to a detector placed at the side of the pipe is noticeably greater than the thickness of the pipe wall.) *For these reasons, HMS4 should be positioned above or below the pipe during piping measurements whenever practical. Moreover, in area source measurements, it appears that the dynamic measurement technique will be superior to static measurements in detecting unobserved accumulation deposits in pipes, since this technique brings the entire surface of the pipe into the detector FOV for some period of time.*

These conclusions will be evaluated by subject matter experts in light of the data provided by the above tests, and recommendations, directions, and quantification of potential errors will be defined in the report of the results of this HMS4 Performance Testing and Validation Plan (PTVP).

Table 5.4. Model variability results

Instrument Identification: _____

Point Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.5])		
%RSD _____ % (equation [5.3])		
Line Source (Known Source _____ grams U-235)		
Measurement 1	Measurement 2	Measurement 3
_____ grams U-235	_____ grams U-235	_____ grams U-235
Mean Measured Value _____ grams U-235 (equation [5.1])		
%R _____ % (equation [5.6])		
%RSD _____ % (equation [5.3])		

5.3 OPERATOR PERFORMANCE VARIABILITY

The purpose of this set of tests (detailed in Table 5.5) is to evaluate the variability of results from a single operator and from different operators when applying the dynamic (area source) measurement technique to pipes. This test requires 5 operators to each perform 14 replicate measurements of the same section of pipe where a known-mass area source has been placed on the inner surface of the pipe. Since this is a controlled experiment to detect differences in measurements of a single operator and among multiple operators, the primary data of interest is the gross counts in the ROI of the 186 keV gamma (region 4) of U-235. The background will be ignored in the statistical analysis since the test environment is a low background area and we are looking for variability in the gross (foreground plus background) measurements.

Table 5.5. Operator performance variability test requirements

Performance Test	Performance Method	Test Results
<p>Determine the variability in measurement results due to operator repeatability and different operator technique. Analyze the data to determine variability in measurement data as a result of single operator variation in technique, and variability between operators.</p>	<p>Confirm operational readiness of the detector MCA pair Identify and use qualified measurement standard(s) Perform area source comparison</p> <ol style="list-style-type: none"> 1 Position the HMS4 detector to emulate standard holdup measurement protocols 2 Place a well characterized area source standard in a surrogate carbon steel >3-in diameter pipe. Model the measurements as an area source by following FSCG-WI-13 3 Operators 1-5 shall perform a measurement using the dynamic quantitative measurement technique applied from the top of the pipe 4 Calculate and report the measured value as shown in Table 5.6. Include the ROI report for full evaluation of the data <p>Note: Operators 1-5 shall perform individual and independent measurements of the same surrogate configuration in sequence</p> <ol style="list-style-type: none"> 5 Repeat steps 2-5 until each operator has made 14 measurements 	

* Since piping measurements are usually carried out in one foot sections at K-25, K-27, the point line, and area source standards should be placed in a known one foot section of the surrogate pipe.

5.3.1 Single Operator Variability in Dynamic Area Source Measurements

To determine a representative measurement uncertainty for a single operator's manipulation of the HMS4 instrument, the variance of each operator will be calculated and summed in quadrature to get an average variance for all five operators. Thus,

$$x_{i,mean} = (1/N_i) \sum_{k=1}^{N_i} x_{ik} \quad (5.7)$$

where, $x_{i,mean}$ is the mean counts for operator i .

$N_i=14$ in this case.

x_{ik} are the individual replicate counts for operator i .

The variance of operator i 's counting over 14 replicates is

$$\sigma_{x_{i,j}}^2 = \{1/(N_i-1)\} \sum_{k=1}^{N_i} (x_{ik} - x_{i,mean})^2 \quad (5.8)$$

and the single operator variance over all 5 operators in the test is

$$\sigma_{x_o}^2 = (1/4) \sum_{i=1}^5 \sigma_{x_{i,j}}^2 \quad (5.9)$$

where, the quadrature is divided by 4 since we will use up one degree of freedom in calculating the mean of all the observations as

$$x_{mean} = (1/5) \sum_{i=1}^5 x_{i,mean} \quad (5.10)$$

The percent relative standard deviation over all operators is then

$$\%RSD = 100 \sigma_{x_o} / x_{mean} \quad (5.11)$$

and the contribution of the variance of measurements by a single operator to TMU for an HMS4 field measurement is

$$\alpha_{oi}(\text{grams U-235}) = \%RSD (m_{meas})/100 \quad (\text{used in Equation [5.19]}) \quad (5.12)$$

where,

m_{meas} is the U-235 mass value in the field HMS4 measurement.

Table 5.6. Single operator performance variability results

Instrument Identification: _____

(Net Counts in ROI 4)					
Test #	Operator # 1	Operator # 2	Operator # 3	Operator # 4	Operator # 5
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
Mean Counts (Equation [5.7])					
Variance (Equation [5.8])					

Variance over 5 operators (Equation [5.9]) _____

Mean over 5 operators (Equation [5.10]) _____

%RSD over 5 operators (Equation [5.11]) _____

5.3.2 Inter-Operator Variability

To answer the question, "Are the techniques of the various operators statistically different?" the student's t-Test will be utilized. Each operator will be tested against each of the other four operators, resulting in a total of 10 tests for significance of difference between the replicate populations.

The t-Test parameter between operator i and operator j is

$$t = (x_{i,mean} - x_{j,mean}) / (\sigma_{x_{i,j}}^2 / N_i + \sigma_{x_{i,j}}^2 / N_j)^{1/2} \quad (5.13)$$

In this case, N_i and N_j are both 14, and $x_{i,mean}$, $x_{j,mean}$, $\sigma_{x_{i,j}}^2$, and $\sigma_{x_{i,j}}^2$ are as defined above.

For this test, (see Table 5.7) values of $t > 1.71$ indicate that there is at least a 90% chance that the difference between the two operator measurement populations is statistically significant.

If all the operators pass all the t-Tests, then this contribution to total measurement uncertainty will be ignored. If not, then the variance between operators will be calculated in quadrature as

$$\sigma_{op}^2 = (1/4) \sum_{i=1}^5 (x_{i,mean} - x_{i,mean})^2 \quad (\text{counts}) \quad (5.14)$$

The percent relative standard deviation (counts) due to variability between operators is then

$$\%RSD_{op} = 100 \sigma_{op} / x_{i,mean} \quad (5.15)$$

and the contribution to total measurement uncertainty used in Equation (5.19) is

$$\sigma_{op} (\text{grams U-235}) = \%RSD_{op} (m_{meas}) / 100 \quad (5.16)$$

where, m_{meas} is the U-235 mass determined in an HMS4 field measurement.

Table 5.7. Inter-operator variability results

Instrument Identification: _____

(t-Test Parameters (Equation [5.13]) (t > 1.71 Indicates Different Population @90% Confidence)					
Operator	Operator # 1	Operator # 2	Operator # 3	Operator # 4	Operator # 5
1	X				
2	X	X			
3	X	X	X		
4	X	X	X	X	

Inter-operator Variance (Equation [5.14]) _____

Inter-operator %RSD (Equation [5.15]) _____

5.4 TOTAL MEASUREMENT UNCERTAINTY

5.4.1 Sources of Measurement Uncertainty

Random and systematic measurement uncertainties are propagated to derive a TMU. Systematic error in a measurement is a consistent and repeatable bias or offset from the true value. Random and pseudo-random errors in a measurement are the identifiable variations between successive measurements made under apparently identical measurement conditions and may include counting uncertainties as well as errors related to ill-defined data reduction parameters, errors in physical measurements, inhomogeneities, matrix interferences, and variations in chemical composition of deposits which may not be defined directly by holdup measurements results. Pseudo-random errors can create either positive or negative bias, and do not display a significant predominance of one type of bias or the other. TMU is determined using both empirical data collected during tests of the instrument measurement capabilities influenced by ambient conditions and modeled performance data that extrapolates for measurement conditions that cannot be readily simulated. Random and pseudo-random error components are added in quadrature. Systematic error components which may create a negative bias on the computed U-235 values are added to the overall estimate arithmetically. A systematic error bias that creates only a positive bias on the computed U-235 values is ignored in the calculation of TMU for confidence level determinations (e.g., "95% confidence").

The contributing uncertainties can be large and numerous. The listing below summarizes uncertainty contributions in a somewhat subjective order of decreasing importance (NRC 1991b).

- (1) Unknown material distribution or location, which affects the source-to-detector distance and the validity of the chosen physical model algorithm, either a point, line, or area source calibration and data reduction
- (2) Self-absorption in the deposit material or its matrix
- (3) Gamma-ray attenuation by intervening matrix materials
- (4) Background interference from distant line-of-sight objects or from adjacent unresolved material
- (5) Detector instability or improper calibration
- (6) Unrepresentative calibration standards
- (7) Counting statistics
- (8) Uncertainty of holdup material isotopic composition
- (9) Deviation of actual measurement geometry from the calibration geometry

Each of the potential contributions noted above has been evaluated in light of the physical, chemical, and isotopic conditions expected to be encountered during the disassembly of the K-25 and K-27 Bldgs. Application of the HMS4 system and its potential measurement errors and uncertainties has also been evaluated in light of the governing technical document (Russo 2005) on which the HMS4 measurements and data reduction algorithms are based.

Many potential sources of measurement error in application of the HMS4 system have been identified. Each of these must be addressed in one of the following ways:

- (1) The error is significant and random (e.g., counting statistics), and will be computed and added in quadrature to the TMU.
- (2) The error is significant and pseudo-random (e.g., measurement of the distance from the source to the detector in calibration), and will be computed and added in quadrature to the TMU.
- (3) The error may have a systematic negative bias on the measurement of U-235 results (e.g., use of process gas enrichments late in the life of the cascade, which would represent the highest possible enrichment deposits and thus the lowest self-attenuation calculations), and a reasonable conservative bounding assumption can be made and utilized in the measurements and data reduction. In this case, the bounding assumption is contained in the nominal measured value and will not be added to TMU.
- (4) The error may have a systematic positive bias on the measurement of U-235 results (e.g., neglecting corrosion and erosion of pipe wall thickness in the calculation of matrix attenuation), and will be ignored as this represents a bounding conservative assumption in the calculation of U-235 mass as described in item (3) above.
- (5) The error may have a random or systematic negative bias on the measurement of U-235 results, but evaluation of the magnitude of possible errors demonstrates that the error is negligible in the HMS4 application in the K-25 and K-27 Bldgs.
- (6) The error may have a systematic negative bias on the measurement of U-235 results, and a reasonable calculation of the potential error can be accomplished for each measurement. This systematic error will be added arithmetically to the TMU for the measurement.
- (7) The error may be systematic and significant, but will be detected in calibration or field measurements, and the causal factor(s) will be corrected and the measurement repeated.

5.4.2 Calculation of HMS4 Total Measurement Uncertainty

5.4.2.1 Calibration Uncertainty

Analysis of the calibration process reveals only two dominant sources of calibration error contribution to TMU. This results from the fact that the U-235 material itself is well characterized in its chemistry, isotopic components, and isotopic masses. However, the source is composed of the uranium material encased in a matrix of materials to allow for handling and positioning, and to assure that there is no loss of material from the source. Thus, the likely sources of error are:

- Computations of self-attenuation within the active source and its containing matrix
- Positioning of the source with respect to the detector geometry during calibration
- Counting statistics

5.4.2.1.1 Source Matrix Self-Attenuation

This error results from pseudo-random inaccuracies in the thicknesses of the source material and the source matrix in which it is contained. Since this parameter could be a significant source of calibration error, it is assumed that great care is taken in utilizing uniform materials and determining their thickness. On this basis, a plus or minus 5% potential error is attributed to these thickness specifications.

Computation of the contribution of this error will be accomplished by increasing the source and matrix material thickness by 5% in the HMS4 calibration algorithm and observing the fractional change in the calibration constant. Since the inferred U-235 content is proportional to this calibration constant, the pseudo-random error will be that same fractional change in the measured U-235 value, which is denoted σ_{sa} , the standard deviation of calibration error due to self-absorption in the calibration source. This error will be combined with other random/pseudo-random errors in quadrature.

5.4.2.1.2 Source Positioning Error

The primary contribution to this error will be errors in measuring the axial distance from the source to the face of the detector. Calculation of this contribution to TMU is described in Sect. 4.2. This error is denoted σ_p , the standard deviation of calibration error due to errors in source positioning. This error will be combined with the other random/pseudo-random errors in quadrature.

5.4.2.1.3 Counting Statistics

Calibration counting statistics for foreground and background measurements will be computed using standard statistical methods and propagated in quadrature with other random and pseudo-random errors.

5.4.2.1.4 Other Potential Calibration Errors

Source positioning error can result from the requirement that the base measurement must be made with the source on the extended axis of the detector/collimator assembly, and the radial measurements must be made at known intervals to the "left" and "right" of the axis of the detector/collimator assembly along a line perpendicular to that axis. If any of these requirements are violated with any significance, a non-symmetric radial response will result. If a nonsymmetric radial response is observed, the geometry of the calibration setup must be corrected, and the calibration reperfomed.

Non-uniform response of the detector/collimator assembly to off-centerline calibration source locations as the detector is rotated on its axis can result from NaI crystal damage or non-uniformity, or misalignment of the collimator/detector assembly. This potential error will be diagnosed by performing a second line-source calibration, with the detector rotated 90 degrees on its axis from the first line-source calibration. If the radial response at either location is noticeably non-symmetric, or if the full-width at half maximum of the two radial responses differ noticeably, the cause must be diagnosed and corrected, and the calibration reperfomed.

Instabilities in instrument gain and linearity are monitored by evaluating the signal from the resident Am-241 source integral to the detector. Any instability will be revealed as part of each measured gamma-ray spectrum. Thus, errors due to instrument instability will be remediated in process, and are ignored in this treatment of TMU.

5.4.2.2 Errors in Specification of Enrichment Levels

The calculation of holdup source self-attenuation by the HMS4 system is most sensitive to the specification of the enrichment level of the deposit. Source self-attenuation is mostly caused by the heavy uranium nucleus due to its very high mass attenuation coefficient (Russo 2005). Total uranium content is determined by dividing the U-235 measured mass by the enrichment. Thus, for highly enriched uranium, the total uranium content in the holdup source may be only about 1.09 times the U-235 mass, while at the lowest enrichments, the total uranium mass may be 100 times the U-235 mass. Moreover, the effect of self-absorption is not linear, but exponential with the inverse of enrichment.

Russo (Russo 2005) calculates that self-absorption is negligible below total uranium areal densities of about 0.2 gms/cm². At very high enrichment, this areal density results in U-235 amounts above the DQOs (and thus the action levels) for U-235 in pipe and other components measured by HMS4. At low enrichments, however, DQO levels of U-235 in pipe result in massive amounts of self-absorption.

Deposits laid late in the life of the cascade would be expected to be at enrichment levels characteristic of process gas late in the life of the cascade. Consumption deposits which may have occurred continuously over the life of the cascade would have an average enrichment equal to the mass flow averaged enrichment at that point in the cascade over the life of the cascade. Deposits laid early in the life of the cascade would have enrichment characteristic of process gas early in the life of the cascade.

Periodic cleaning of process piping and equipment would tend to cause deposits to be at the higher end of enrichment possibilities. Since a majority of the deposits that HMS4 is utilized to detect probably occurred continuously over the life of the cascade, the appropriate nominal assumption might be to use the mass flow averaged enrichment at any point in the cascade. However, this assumption would still be subject to measurement uncertainty since any given deposit could have been laid earlier in the life of the cascade, and remained undisturbed thereafter. There appear to be two choices here. The first would be to compute a custom-tailored measurement uncertainty for each measurement location. This would introduce field operational complexities that are unjustifiable in light of the precision benefit realized. Alternatively, a bounding conservative assumption could be utilized for the deposit enrichment at any point in the cascade for the nominal U-235 mass calculation. To account for deposits that may not be representative of continuous consumption over the life of the cascade, and might have been laid, at least partially, early in the life of the cascade, the assumed deposit enrichment at any point in the cascade will be taken as 50% of the mass flow averaged process gas enrichment over the life of the cascade at a given measurement point in the cascade. For this component of TMU, the uncertainty is embodied in the nominal calculation, so no contribution to TMU will be calculated.

5.4.2.3 Gamma-Ray Attenuation by Matrix Materials between the Source and the Detector

The material configurations originally installed in items that are measured by the HMS4 system are well characterized and known. The major error contribution is expected to be material corrosion and erosion over time that has reduced the matrix attenuation compared to the model used in the HMS4 data reduction algorithm. This creates a positive bias on measured U-235 amounts. Thus, this effect will be ignored in the computation of TMU for HMS4 measurements.

5.4.2.4 Holdup Source Location and Distribution

The large majority of HMS4 measurements at K-25 and K-27 are carried out on piping systems. It is generally expected that undisturbed holdup deposits will be uniformly distributed. However, if the deposit has been exposed to moist air, oxidation and hydration of the deposit have been observed to occur, with the hydrated oxides becoming loose and falling to the bottom of the pipe in question. Since all HMS4 measurements (either area source measurements or finite line source measurements) assume uniform deposits on the inner surface of a pipe, an error can be introduced if this "oxidation-hydration-separation" phenomenon has occurred. The bias created by this error can be either positive or negative, depending on the measurement technique utilized and the source-detector geometry for a specific measurement. This potential error is mitigated by the VPD program in which the interior of the pipe is visually observed via bore scope, and piping segments with visible deposits are removed from the system.

For both the dynamic measurement technique using an area source algorithm and the finite line source measurement technique, tests are specified in this document to define the effect on these measurements of the source being concentrated in the bottom of the pipe. The results of those tests will be used to define a

contribution to total measurement error to be attributed to this phenomenon. The exact nature of this error parameter, denoted σ_s , will be defined after the test results are known.

5.4.2.5 Approximation of the Curved Surface of a Pipe as a Plane

Measurement of the interior surface of a pipe using the area source algorithm incorporates the assumption that the curved surface of the pipe can be approximated as a planar area. This assumption introduces two potential errors.

First, the detector FOV of the interior surface of the pipe encompasses more of the area source at a given FOV than is assumed in the data reduction algorithm. This will create an unknown (at this time) positive bias on U-235 mass results.

Second, unattenuated U-235 gamma rays will pass through a slightly longer track length in the source material and in intervening matrix attenuation materials before encountering the detector. Thus, both self-attenuation and matrix attenuation will be greater than reflected in the data reduction algorithm. This error will have a negative bias on U-235 mass results.

Direct experimental quantification of these compensating biases, either separately or together, will be difficult due to the presence of other error inducing phenomena in measurements. Quantification of the composite effect of these two error terms will be accomplished via comparison of Monte Carlo simulations of planar geometries and the actual curved geometry of piping configurations. This error will be systematic, and could be either a positive or a negative bias on U-235 measurements. Since the errors compensate one another to some extent, the magnitude of the composite error may be negligible. If significant and a negative bias on U-235 results, it will be denoted e_{pi} , the systematic error induced by the planar assumption, and added arithmetically to the TMU. If the bias is positive for U-235 results, this error will be ignored in the computation of TMU as it would represent a conservative bounding assumption.

5.4.2.6 Background Measurement Errors

In situ measurements of holdup material can be significantly influenced by ambient background emanating from other sources in the area, or sources external to the building. HMS4 protocols require that, where possible, the geometric configuration of a measurement be executed in such a way as to minimize background contributions from obvious adjacent sources (pipes and equipment). Moreover, attenuation of background by the target matrix is accounted for in the HMS4 data reduction algorithm.

Systematic errors in background measurements arise due to the fact that the detector is moved for the background measurement, maintaining the direction vector of the detector axis while removing the foreground target from the detector FOV. Thus the FOV of the background measurement is slightly altered. With this in mind, the systematic component of background measurement will be pseudo-random, and defined as plus or minus 15% of the measured value, denoted σ_{het} , the standard deviation of the background measurement due to pseudo-random errors in the background measurement. This parameter must then be corrected for additional attenuation in the target matrix that would have been encountered in the FOV of the foreground measurement. (This is a situation-specific correction as discussed in Sect. 2.2 of this document.) This component of TMU will be combined with background and foreground counting errors in quadrature as follows:

The corrected count rate for observation of a pipe or other object in an area measurement is

$$cCR_B = (A_4 - A_5) / T_A - (B_4 - B_5) / (T_B * tt) \quad (5.17)$$

where,

cCR_B is the corrected count rate in ROI 4

A_4 is the total (foreground plus background) counts in ROI 4

A_5 is the total (foreground plus background) counts in ROI 5

B_4 is the background counts in ROI 4

B_5 is the background counts in ROI 5

T_A is the counting time for the total counts

T_B is the background counting time

tt is the correction factor for attenuation by materials that attenuated the background during the foreground measurement (e.g. two pipe wall thicknesses).

The variance of the corrected count rate is

$$\sigma_{cCRB}^2 = (A_4 + A_5) / T_A^2 + B_5 / (tt * T_B)^2 - (B_4 + .0225B_4^2 + B_5 + .0225B_5^2) / (tt * T_B)^2 \quad (5.18)$$

This error will be propagated with other random-pseudo-random errors to define the TMU contribution from background measurements.

5.4.2.7 Computation of the Total Measurement Uncertainty

The variance in the calculated value for U-235 will be the sum of the variances of all the contributing parameters with random and pseudo-random errors.

$$\sigma_{U-235}^2 = \sigma_{c_1}^2 + \sigma_{c_2}^2 + \sigma_{c_3}^2 + \psi^2 \sigma_{cCRB}^2 + \sigma_{w_1}^2 + \sigma_{w_2}^2 \quad (5.19)$$

where,

σ_{U-235} is the standard deviation of the measured U-235 value.

ψ is the conversion factor from net counts per unit time to grams of U-235, including detector efficiency, matrix attenuation, self-absorption, and finite width point or line source correction.

$\sigma_{w_1}^2$ and $\sigma_{w_2}^2$ are defined as zero for all but the dynamic measurement technique.

Note that this formulation assumes that counting statistics during calibration are negligible.

The TMU will thus be

$$TMU = (\sigma_{U-235}^2)^{1/2} + c_{95} \quad (5.20)$$

and the 95% confidence level result be the nominal measurement plus 1.645 times the TMU to get 95% confidence in one-tailed statistics.

5.5 LOWER LIMIT OF DETECTION AND MINIMUM DETECTABLE AMOUNT

The Lower Limit of Detection (LLD) is defined as the net signal level above which an observed signal may be reliably recognized as detected, whereas the MDA is dependent upon ambient interferences in the location of measurement. Lower Limit of Detection and MDA are inextricably linked since LLD defines, for lack of a better word, system "noise" while MDA defines ambient "noise" that is above the identified "noise" indigenous to the instrument. In a stable environment LLD and MDA would be expected to be identical; however, field measurements, performed in situ, are complicated by ambient factors that cannot be specifically ascribed to material holdup in the measurement location. A predominant influence on the MDA is ambient background. For a typical in situ measurement, a background measurement is performed in the general area of the component without including the deposit being assayed. This is done to determine the contribution of gamma rays present within the detector FOV not ascribed to the deposit. In situ background measurements are not performed by removing the component or by removing the deposition of radioactive material from the component. Current in situ measurements are performed in the K-25 Bldg. where there is a sizeable background contribution due to the distribution of material holdup.

Although the sensitivity for nondestructive waste assay systems is generally quoted as three sigma over background, the more widely accepted approach is based on "Limits for Qualitative Detection and Quantitative Determination" (Currie 1968). See also "The Minimum Detectable Activity Concept" (Lochamy 1981), "IEEE Transactions on Nuclear Science" (Walford, et. al. 1971) and EG&G Application Note 17 (Lochamy 1981).

From the position paper and the above references, two principal limiting levels are defined:

- (1) Decision Level or Critical Level (L_C)—the net signal level above which an observed signal may be reliably recognized as detected.
- (2) Lower Level of Detection (L_D)—the lowest practical *quantity* that the instrument can routinely and reliably detect. "Routine detection means that if the activity is equal to the detection limit, the instrument will report a detected result at least 95% of the time, but not necessarily equal to the detection limit."

For a 95% confidence level for one-tailed statistics, the probability of incorrect positive assumption (Type I error, α) and an incorrect non-detection (Type II error, β) are set equal to 0.05. This was the standard adopted by Martin Marietta Energy Systems, Inc. at the time. The decision or critical level equation is based on the maximum allowable value for α and the standard deviation of the net signal when its limiting mean is zero. The equation for the decision level is given by:

$$L_C = k_\alpha \cdot \sigma_0 \quad (5.21)$$

where,

k_α = abscissa of the standardized normal distribution corresponding to the probability level $1-\alpha$.
For 95% CI and one-tailed statistics, $k = 1.645$;

σ_0 = standard deviation when the limiting mean of the net signal is zero;

The detection limit is based on the value determined for L_c , the maximum allowable value for a Type II error (β), and the standard deviation of the net signal when the limiting mean is equal to L_D . The detection limit equation is defined as:

$$L_D = L_c + k_\beta \cdot \sigma_D \quad (5.22)$$

where,

k_β = abscissa of the standardized normal distribution corresponding to the probability level $1-\beta$.
For 95% confidence level and one-tailed statistics, $k = 1.645$;

σ_D = standard deviation when the limiting mean of the net signal is L_D ;

For the case of an active assay in HMS4 where the background is much greater than the net signal, the variations in the standard deviations from a net signal of zero to L_D are slight. Therefore, the standard deviation of the net signal can be assumed constant, i.e., $\sigma_D = \sigma_0 = \sigma$.

For the variance of a net signal given by:

$$\sigma^2 = \sigma_{s+b}^2 + \sigma_B^2 \quad (5.23)$$

where,

σ_{s+b}^2 = variance of the "sample + blank" signal;

σ_B^2 = variance of the blank signal;

this assumption results in the variance being equal to twice the blank variance or

$$\sigma^2 = 2 \cdot \sigma_B^2 \quad (5.24)$$

If the standard deviation of the blank has been made negligible due to multiple observations as in the case of HMS4, the variance of the net signal is simply equal to the variance of the blank. For paired observations where the background is not known well, the standard deviation will differ from the case of the well known blank by the square root of two.

If the risk of 5% for a Type I or Type II error is acceptable (95% confidence level for one-tailed statistics), the following equations for the decision and detection limits are valid for active measurements in HMS4:

$$L_c = 1.645 \cdot \sqrt{2 \cdot \sigma_B^2} \quad (5.25)$$

$$L_D = 1.645^2 + 2 \cdot L_c \quad (5.26)$$

For reporting the results for HMS4 MDA,

the LLD is the counts measured by the detector which are statistically measurable above the background counts. The LLD is dependant on the background measurement used for each location. The sum of the analysis peak and the continuum from the background spectrum is used to calculate the LLD. Using the following equation,

$$LLD = k^2 + 2k\sqrt{2*(B_{ROI1} + B_{ROI2})} \quad (5.27)$$

where,

k is 1.645 which corresponds to a 95 % confidence level

B_{ROI1} is the value of the analysis peak in background spectrum

B_{ROI2} is the value of the continuum in the background spectrum

Note also that in the case of component measurements using the area source algorithm, the background measurements must be corrected for the attenuation of the target component as described in Fig. 2.3 before the LLD is calculated.

The MDA is calculated using the LLD calculated above, the count time, and the correction factor for the attenuation of the container wall. Using the following equation,

IF the source type is a Point **THEN**,

$$MDA = \frac{LLD * K_s * CF_{wall} * r^2}{T} \quad (5.28)$$

ELSE IF the source type is a Line **THEN**,

$$MDA = \frac{LLD * K_s * CF_{wall} * r}{T} \quad (5.29)$$

ELSE IF the source type is an Area **THEN**,

$$MDA = \frac{LLD * K_s * CF_{wall}}{T} \quad (5.30)$$

where,

LLD is the counts calculated above

K_s is the calibration constant for a point, line or area source

T is the counting time in seconds

CF_{wall} is the correction factor for the wall material

r is the detector standoff

Table 5.8 defines a set of tests to determine MDA for point, line and area sources in the low-background laboratory environment

Table 5.8. LLD test requirements

Performance Test	Performance Method	Test Results
<p>Establish the Lower Limit of Detection for the HMS4 as defined in Russo (Russo 2005) and ORNL (ORNL 2007)</p>	<p>Confirm operational readiness of the detector MCA part</p> <p>Use a surrogate component that is representative of the normal (average) population of pipe (carbon steel - schedule 40)</p> <p><i>Perform blank measurements</i></p> <ol style="list-style-type: none"> 1 Utilizing an appropriate detector holder or hardware apparatus setup a surrogate component that is representative of actual measurement configuration for which the HMS4 measurements are anticipated to be applied. Model the measurements as a point source by following E SG-WI-13 2 Position the HMS4 detector to emulate standard holdup measurement protocols 3 Perform 15 replicate measurements of the surrogate configuration 4 Calculate and report the measured value. Include the ROI report for full evaluation of the data 5 Reanalyze measurements modeling as a line and then an area 6 Calculate and report the measured value for both line and area models. Include the ROI report for full evaluation of the data 7 Calculate L_C and L_D as described in Sect. 5.5 8 Report 'pristine' Instrument LLD and use calculations from Sect. 5.5 to calculate MDA 	

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6. QUALITY RECORDS

6.1 QUALITY RECORDS GENERATED

The Quality Records listed below, generated under this PTYP, document that both the instrument and method are qualified for specific NDA measurements.

6.1.1 Quality Records for Instrument Qualification

- **Data sheets for all measurements.** Data tabulated using the forms provided in this document or other BJC forms used to collect calibration data.
- **HMS4 Dump report of all measurements.** HMS4 generated Crystal Reports listing specific details of each measurement taken, date, time etc. including backgrounds and source check measurements.
- **HMS4 ROI report of all measurements.** HMS4 generated Crystal Reports showing number of raw counts for all five ROIs.
- **HMS4 Analysis report of all measurements.** HMS4 generated Crystal Reports listing specific details of backgrounds, foregrounds, finite source correction factors, self-attenuation corrections factors, and U-235 mass.
- **HMS4 totgms modified of all measurements.** HMS4 generated Crystal Reports listing measurement ID, type of source, U-235 mass, measured value, MDA and reported value with any uncertainty.

6.1.2 Quality Records for Method Qualification

- **Data sheets for all measurements.** Data tabulated using the forms provided in this document.
- **HMS4 Dump report of all measurements.** HMS4 generated Crystal Reports listing specific details of each measurement taken, date, time etc. including backgrounds and source check measurements
- **HMS4 ROI report of all measurements.** HMS4 generated Crystal Reports showing number of raw counts for all five ROIs.
- **HMS4 Analysis report of all measurements.** HMS4 generated Crystal Reports listing specific details of backgrounds, foregrounds, finite source correction factors, self-attenuation corrections factors, and U-235 mass.
- **HMS4 totgms modified of all measurements.** HMS4 generated Crystal Reports listing measurement ID, type of source, U-235 mass, measured value, MDA and reported value with any uncertainty.

6.2 QUALITY RECORDS MAINTENANCE REQUIREMENTS

Maintain records in accordance with BJC-OS-1001, "Records Management Including Document Control" (BJC 2008).

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7. REFERENCES

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- BJC 2008. "Records Management, Including Document Control." BJC-OS-1001, Bechtel Jacobs Company LLC, Oak Ridge, TN.
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- Russo, P.A. June 2005. "Gamma Ray Measurements of Holdup Plant-Wide: Application Guide for Portable, General Approach" LA-14206.
- Walford, G. V., D. Aliaga-Kelley, and W. Gilboy 1972. *IEEE Transactions on Nuclear Science*, Vol. NS-19.

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8. SOURCE DOCUMENTS

1. **Principal emissions and primary data for U-234, U-235, and U-238**
2. **Attenuation Data for Steel and Uranium**
3. **Detection Efficiency Data for Na (TI) Crystal 0.5 inches thick and 1.0 inch diameter**
4. **Example Calibration Test Report**
5. **Example Measurement Test Report**
6. **FSG-WI-13, HMS4 Holdup Measurements**
7. **BJC-DE-0716, "NDA Training and Qualification"**
8. **BJC-KD-8311, "NDA Data Quality Assessment for the K-25/K-27 D&D Project"**

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BJC/OR-2930/R1

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Hanson, Nancy J (3NH)

From: Hughes, Vickie J (VMH)
Sent: Tuesday, April 15, 2008 3:01 PM
To: Howard, Jack L. (Q8H)
Cc: Carlson, John D (J44); Deacon, Karen E. (K7A) ; Detorre, Benjamin J (DTE); Fillers, Fredrick Donald (FFI); Howe, Kathleen E (HKF); Mccague, James (Jim) (MGI); Rigsby, Virginia P (VLR); Royce, Ralph Robert (VRH); Shaffer, Karen E (KRN); Shelton, Michael J. (U4M); Thiesing, James W (TIX); Tunstall, Mickey P (MYT); EMEFDMC; K25/K27 PDCC; 'Bill.Riley@orau.org'; 'Jeff.Chapman@orau.org'
Subject: K25-08-071 Performance Testing and Validation Plan for HMS4 Quantitative Gamma Measurements (BJC/OR-2930/R1)
Attachments: K25-08-71 Trice to Howard.PDF; BJC OR-2930 - FINAL-FINAL- 4-14-08.pdf

The subject letter and enclosure are being mailed to Jack Howard.



K25-08-71 Trice to BJC OR-2930 -
Howard.PDF ... FINAL-FINAL- 4-1...

Thanks,
Vickie Hughes

K25/27 Project
Building: K-2527-R Room 15
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