

# **Optimum Method to Determine Gamma Activity in 200 Liter Drums – *In-toto* Measurement or Extracting a Sample**

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**Abstract.** In the process of decommissioning contaminated facilities, and in the conduct of normal operations involving radioactive material, it is frequently required to measure radioactivity in large containers such as 200 liter drums. For solid material, it is quite unusual for the radioactivity to be homogeneously distributed throughout the container. One way to derive the concentration of radioactivity within the container is to extract a sample for subsequent measurement in the laboratory. Another way is to use gamma spectroscopy and assay the entire container, or *in-toto* measurement. This paper examines the process of determining the best way to estimate the activity within the container, and gives quantitative estimates of measurement uncertainty for various conditions of radioactivity contained within 200 liter drums. When the contents of the container are not homogeneous, the sampling uncertainty is likely to be larger than the *in-toto* measurement uncertainty.

**KEYWORDS:** *Gamma spectroscopy, in-situ, in-toto, uncertainty, drum, sampling*

## **1. Introduction**

A common measurement task is to determine the radioactivity concentration or content within 200 liter drums. If the contents of the container were well known, and if both the matrix within the container and the radioactivity within the matrix is uniform, then this it is a rather conventional task to evaluate the best way to measure it. Extracting a sample for laboratory measurement, or performing *in-toto* measurements of the entire container are well known and common methods.

But in practice, this convenient assumption of homogeneity is rarely the case. In both cases (*in-toto* measurement and sampling followed by laboratory measurement) this non-uniform distribution of radioactivity greatly compromises the accuracy of the assay, and makes uncertainty estimates much more complicated than simple propagation of counting statistics. It is the purpose of this paper to show which of these methods is more accurate, and the appropriate use of each.

*In-toto* quantitative assay of the containers can be performed by a variety of devices ranging from automatic multi-detector scanning rotating systems to simple portable single detector systems. There are many choices of counting protocol that can be made, depending upon the level of accuracy needed, and depending upon the sensitivity and counting time desired. These include: detectors close to the container or far away; single or multiple detectors; fixed detectors or scanning; fixed drum or 180 degree rotation or continuous rotation.

With uniform distribution of radioactivity in a known matrix the efficiency is easily and accurately determined with radioactive sources or with mathematical calibrations. Since calibrations are most always made with uniform distributions, measurements where the radioactivity in the container is not uniform will be in error. For non-uniform distribution of radioactivity, the largest source of uncertainty is likely to be because a uniform reference calibration does not accurately represent the true efficiency. However, certain counting protocols which will be explored in this document can be shown to greatly reduce that uncertainty.

Alternatively, the container may be opened and a sample extracted and sent to the laboratory. If the sample is subsequently homogenized and properly prepared and counted in a geometry where the calibration is well known, the measurement uncertainty should be small. However, the accuracy of the radioactivity in the original container depends upon how well the laboratory sample concentration represents the container concentration. When the radioactivity in the container is not uniformly distributed, the largest source of uncertainty will very likely be the sampling uncertainty. However, certain sampling protocols discussed later can be shown to greatly reduce that uncertainty.

So, given the choice of *in-toto* measurement of a container with an imperfect gamma assay method, or extracting a sample followed by a [assumed perfect] laboratory assay: which will give a more accurate determination of the contents of the container?

## 2. Measurement Objectives

The first step in the process is to find out the objectives of the measurement, and therefore the quality of the measurement that is required. This is a frequently overlooked step in the process. Rarely is the objective as simple as “how much radioactivity is in that drum?”. More likely it is something like “give me enough information to reliably categorize these containers as A, B, or C”.

The project manager planning the measurement campaign must try to find out as much as possible about the desired measurement objectives so he can develop a measurement strategy to best achieve it.

Examples of things to inquire about:

- Desired accuracy of each individual container, or of a group of containers; frequently a function of the level of activity and the type of nuclide
- Decision points where the containers are categorized into the appropriate group
- Acceptable Confidence Level for placing the containers in the correct category
- Timing – when are the results needed
- Cost – how much labor can be expended, and what kind of equipment can be used; the cost penalty for an incorrect decision
- Accessibility – are the drums easily to access, can they be moved, is moving equipment available
- Past measurements or other knowledge – do you know the nuclides to be expected, the range of activity expected, the level of uniformity expected, ...

## 3. Measurement Strategy

The proper development of a measurement strategy can only happen after first knowing the Measurement Objectives, and by then understanding how the uncertainty in the measurement result changes due to the many possibilities of performing it.

The two primary measurement possibilities are *in-toto* measurement of the entire intact container via gamma spectroscopy, and extracting a sample of the container for subsequent laboratory analysis. Each one of these methods has both good and bad points, which the program manager must understand well enough to design the most practical method that meets the agreed-upon Measurement Objectives.

### 3.1 *In-Toto* Measurements

- Generally restricted to only gamma measurements
- Detection levels generally quite good since the sample quantity is large
- Low energy or low abundance gammas may be problematic at low levels
- Can be performed with NaI or LaBr scintillators or Ge semiconductor detectors
- Instruments with poor energy resolution may have interference problems from NORM or other radioactivity in the container or nearby
- Opening of the container is not required
- Instantaneous results can be used to guide future measurements on that container or others
- Expensive and sometimes fragile equipment must be used under field conditions
- Efficiency calibrations of large items problematic if using radioactive sources, but easily done with mathematical methods
- No radioactive waste generated
- Taking multiple measurements of a container from different perspectives can reduce the uncertainty and provide evidence of uniformity, or the lack of it.

### 3.2 Sampling of Container followed by Laboratory Measurements

- Opening of the container is required, with the appropriate level of safety protection
- Obtaining a representative sample from locations other than the top of the container is difficult
- Sample must be packaged and shipped to laboratory
- Chain of custody of sample and matching to the original container must be established
- Can perform chemical separation on sample for removal of interferences and optimum sample geometry for alpha, beta, and gamma assay
- Sample preparation other than for simple gamma spectroscopy is generally rather expensive
- Detection levels can be quite good since sample is close to detector and counting times can be long
- Results not generally available for weeks to months
- Samples must be disposed as radioactive waste
- Taking multiple samples from a container can reduce the uncertainty and if analyzed separately can provide evidence of uniformity, or the lack of it.

### 3.3 Uncertainty Estimation

If the contents of the container are well known and uniform, then the measurement is a simple task. Either use a simple calibration method for the field *in-toto* method, or open the container and scoop some of the contents from the top and take it back to the laboratory. Under these conditions the measurement uncertainty will generally be dominated by counting statistics, which are well understood and easily handled by conventional methods.

For uniform geometries of well-known matrices, efficiency calibrations are rather easy to perform. Mixtures of radioactive calibration sources distributed in a simulated uniform manner have long been used successfully. More recently, mathematical calibrations like MCNP [1] and ISOCS<sup>®</sup> [2,3,4] have been used and accepted by the user community for both calibration of large geometries like here and for laboratory geometries. They are especially convenient to use and when the matrix is something other than water – e.g. soil, concrete chunks, metal shavings, ...

But this idealistic situation of a uniform matrix and a uniform distribution of radioactivity within the matrix is rarely the case. It is unusual for the matrix within a specific container to be well known in composition, but rather it is commonly a mixture of various materials in unknown concentrations where the matrix varies within the container, and where the density is NWK and varies within the container. It is even more unusual for the radioactivity to be homogeneously distributed within the container.

Consequently any efficiency calibration, source-based or mathematics-based, will be wrong when any of these conditions exist. This is true in both laboratory measurements of the sample and *in-toto* measurements. For *in-toto* measurements, this deficiency of the generic calibration used to accurately represent the exact conditions in the container under measurement is most likely the major contribution to the uncertainty of the *in-toto* measurement. For laboratory measurements, the sample preparation normally should transform the sample into the proper form to assure validity of the measurement instrument calibration. However to translate those laboratory results into container results requires propagation of laboratory uncertainty [normally small] and the sampling uncertainty [can be quite large].

For the conditions expected to be encountered, what is the *in-toto* measurement uncertainty? And if samples are extracted, what is the sampling uncertainty ?

To help answer questions like this, a new feature to the ISOCS efficiency calibration software has been developed that allows uncertainty computations to be performed where there is incomplete knowledge of an accurate representation of the sample. The ISOCS Uncertainty Estimator [IUE] software [6, 7, 8, 9] can also simulate various non-uniform distributions in containers, and then compute the resulting uncertainty in the efficiency calibration. The software also has a feature that simulates various sampling

methods. The IUE software can then be used to create various non-homogeneous distributions in 200 liter drums, and then to estimate both the calibration uncertainty and the sampling uncertainty.

### *3.3.1 ISOCS Uncertainty Estimator – Overall Operation*

IUE has been developed to improve the quality of the gamma spectroscopy uncertainty estimate, to improve the ease of generating these uncertainty estimates, and to document how they were generated.

The user first runs the ISOCS efficiency calibration software in the normal manner to compute the normal reference efficiency for the sample being measured. This efficiency file has encoded within it the inherent uncertainty in the ISOCS efficiency calibration method – i.e. 4-8%. As with most efficiency calibrations, this assumes the calibration model is a perfect representation of the sample.

Some of the ISOCS data entry parameters are well known and do not vary appreciably; e.g. the container is always known to be type 304 stainless steel. Other [or most in many situations] parameters are not well known [NWK], e.g. the wall thickness of the container or the density of the contents. It is these “NWK” parameters that contribute to the uncertainty in the calibration efficiency. This is equally true for source-based calibrations and mathematical calibrations. For each NWK parameter, the user is required to provide to the IUE software an estimate as to how much that parameter varies. This can be determined, for example, by measuring a group of containers, or by consulting the manufacturing specifications for the containers, or just by making educated guesses. The parameters that can be varied include dimensional parameters [diameter, distance, thickness, density, ...], as well as material composition of each item in the model.

For each NWK parameter, the user provides upper and lower limits [e.g. maximum and minimum density] and a distribution form that the parameter values within those limits are assumed to follow. As an example of this distribution form, if the values were determined by a series of measurements, then the limits can be assigned to represent 1 standard deviation, 2 standard deviations, or 3 standard deviations. If the values are just known as limits, then they could be assigned a uniform distribution function [all values equally probably] or a triangular distribution function [zero probability beyond the limits increasing linearly to the maximum probability in the middle].

The IUE software then assigns a value to each of these NWK parameters following the probabilities defined by the distribution function for that parameter. The efficiency is computed for that model. This process is repeated a large number of times until adding more models doesn't change the result. The software then computes the model-to-model uncertainty for each energy, which will then be combined with the calibration uncertainty and the counting statistics uncertainty. If the radioactivity within the container distributed in a non-uniform manner, then IUE can be used to examine various non-uniform distributions to estimate that portion of the Total Measurement Uncertainty [TMU].

### *3.3.2 IUE Data Entry Method*

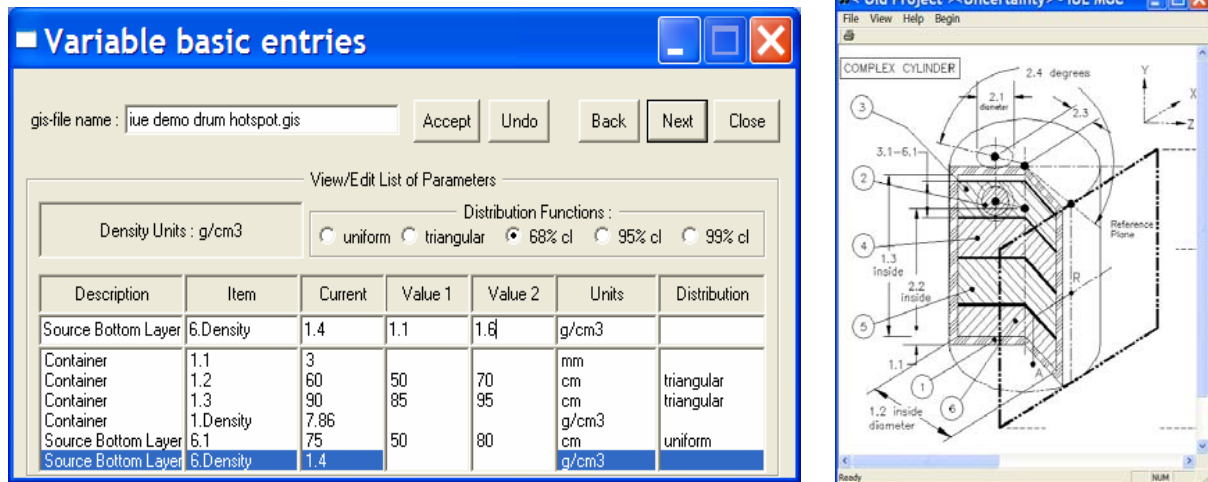
The user first points the software to one of the intermediate files created in the normal process of performing an ISOCS efficiency calibration. This file contains all the physical parameters of the normal [assumed perfect] calibration model.

The user is then presented with a series of screens showing all the parameters from the calibration model, and given an opportunity to make each of them a variable parameter. If that parameter is to be varied, then the user enters for each parameter the minimum value, the maximum value, and the distribution function to be used. Two examples of these screens are shown below as Figures 1 and 2.

In the case where the variable parameter is a material, the user enters a series of discrete materials, along with a weighting factor denoting the likelihood of that particular material being present. All input parameters are stored in a file, and in a printed report for the project record.

### *3.3.3 Computational Methodology*

The method used in this software is Probabilistic – all variables are assumed to vary randomly, but in a manner as described by their individual probability distribution function. All variables [except a few that are noted elsewhere] are assumed to vary independently from others, to the extent that it is physically possible.



**Figure 1:** Typical IUE data input screen (left). Parameters are entered here to describe the amount and type of variation for the model. Entries correspond to the numbers on the graphic on the **Figure 2** (at right).

Using these rules, the IUE software creates the files for a series of ISOCS calibration models. A random process is used to generate values for each NWK parameter, according to the probability distribution function rules and limits defined by the user. These values are combined to create an ISOCS model. A large number of these models are created and checked for validity.

The IUE software then computes the efficiency for a large number of energies using each of the valid random models. The IUE software now contains an array of efficiency values at each energy. For each energy, the IUE software then computes the mean efficiency, and the standard deviation of those efficiency values at that energy. This standard deviation now represents the uncertainty from the combined effect of all the NWK parameters.

This uncertainty is then combined with the basic ISOCS calibration uncertainty and embedded within the efficiency calibration. When this efficiency calibration is used to analyze a sample, this total calibration uncertainty is propagated with counting statistics uncertainty and other uncertainties for the final total measurement uncertainty.

### 3.3.4 Other Software Features

For measurements situations that use multiple detectors, the software allows the user to specify the number of detectors and their placement around the object. Therefore it can be used to calibrate or estimate uncertainty from common field measurement systems like box or truck counters.

For measurement situations that use rotating samples, the software allows the user to specify this, and to define how many discrete steps are used to simulate a continuous rotation. For measurement situations that use scanning detectors, the software allows the user to specify this, and define how many discrete steps are used to simulate a continuous scan. This allows the software to be used to calibrate and estimate uncertainty for common drum measurement systems.

Some measurement situations have non-uniform sample concentration. Several of the ISOCS sample shapes [templates] allow non-uniform distributions, including “hot spots”. The IUE software expands

that by allowing a multiple [or variable] number of hotspots and the size of the hotspots [fixed or variable] to be included in the model.

Although most of the variables are treated as independent variables, a few of them can be inter-dependent. A common example is sample height in a container, sample density, and sample weight. The weight is typically the most well known parameter, as it is rather easy to determine. The IUE software lets the user enter the weight as a variable parameter and then it computes either sample height or sample density.

The software computes the arithmetic mean efficiency and standard deviation, as well as the geometric mean efficiency and standard deviation. For measurement situations where attenuation is the dominant factor, the values are more likely to be in a log-normal pattern, where the geometric values are more relevant.

The IUE software also operates in a Sensitivity Mode, where only 1 parameter is varied at a time. This provides the user with feedback as to which of the parameters are the major contributors to the total uncertainty, thus allowing the user to concentrate data collection resources on those dimensions that are most important.

The IUE software can also simulate the extraction of a portion of the container and compute the “representativeness” or uncertainty of that sample. This process only works on those containers that have “hot spots” of non-uniform radioactivity. The user specifies the sample type [core or grab], specifies the sample volume to be extracted and how many sub-samples will make up the total sample extraction volume. The IUE Sampling program then simulates extraction of a these samples from the same containers that were used for the previously described *in-toto* uncertainty analysis. This done multiple times using random sampling locations from each of the containers. The software computes the fraction of the radioactive source within each sample as compared to the amount in the container. Then the uncertainty is computed from the total population of samples.

#### **4. 200 Liter Drum Assay Uncertainty Calculations**

##### *4.1 Homogeneous Distribution Example*

An in-situ Ge gamma spectroscopy system is being used to assay a group of 200 liter drums filled with radioactive soil from an environmental remediation project. The site had contaminated soil of many different types, and consequently many different densities. These ranged from wet sandy material at density of around 1.8 g/cc down to soil mixed with decayed vegetation at densities of 1.0. The soil composition of the containers was estimated to be normal soil approximately 50% of the time, mostly sand approximately 25% of the time, and soil and decayed vegetation about 25% of the time. The drums were filled from material that had been stored in piles and are rather well mixed, therefore it is reasonable to assume that the radioactivity in each individual container is homogeneous.

The composition and density of each individual drum is not known, but the total weight of each drum is known. The weight of the containers varied from 400 to 800 lbs. A random sampling of the weights showed that 95% of them were between 450 and 750 lbs.

The fill height of each drum is not known, and it is neither practical nor desirable to open each drum for inspection. But from discussions and procedures during the fill operations, the containers were filled until they were approximately 70-90% full.

The ISOCS cart and detector were wheeled up next to the drums, at approximately 100cm from the side of the drum. That distance was measured, and the cart repositioned if the distance was not between 90 and 110 cm. The detector in the ISOCS cart is 26 cm from the ground, and aimed at the center of the drum. But since the ground is not flat, there could be a 10cm variation in the detector height.

The drum specifications from the manufacturer claim that the diameter and height of the container are controlled to within 1cm of the nominal value, and the wall thickness is controlled to within 20% range.

The nuclides of interest for this site are Am-241 at 60 keV, Uranium-235 at 185 keV, and Uranium-238, using the Pa-234m daughter at 1001 keV.

There are 8 uncontrolled variables in this problem. What uncertainty should be assigned to the combination of all these variables when counting an individual drum? To answer this question the above data were used with IUE in the Uncertainty Analysis mode. The program created several hundred mathematical calibrations which were analyzed for standard deviation. Table 1 shows the 95% CL uncertainty estimate. The first row in the data is when all the parameters were allowed to vary. From the IUE Sensitivity Analysis results [not shown here], the user knew that density was a big factor, and wanted to hypothetically explore what would happen if he would more accurately determine it. The next row shows the result. Still not satisfied, the user suspected that the steel drum wall would adversely affect the Am241 uncertainty, and evaluated the use of an ultrasonic probe to accurately measure the wall thickness, which removes that variable and gives the results in the last row.

<b>Table 1 Uncertainty analysis results for 200 liter drum</b>		
<b>Condition</b>	<b>95% CL at 60keV</b>	<b>95% CL at 1001keV</b>
All items variable	36%	20%
After fixing the density	30%	14%
After fixing the container wall	16%	14%

#### *4.2 Non-homogeneous distribution of radioactivity in 200 liter drums – in-toto measurement*

This exercise will illustrate the usefulness of the IUE software to optimize a counting geometry, and then to assign an uncertainty to the efficiency calibration for that optimum geometry. In this scenario, there exist a large number of 200 liter drums filled with soil, at an average density of 1.2 g/cc. The radioactivity in the soil is known to be quite non-uniform. The radioactive soil is contained in grapefruit-sized nodules [hotspots] which are interspersed in non-radioactive soil of the same composition and density. The nuclides of interest have energies of 60 keV and 1000 keV. What is the optimum counting geometry if the purpose is to minimize the total uncertainty of the drum assay?

The largest contribution to the uncertainty is the number and location of the radioactive hotspots in the drum. Therefore all other items were considered “well-known” and were not varied. The variables were simply the number of radioactive sources per drum. Situation one assumed that there were 1-5 radioactive hotspots per drum, all values equally probably, and all sources randomly distributed. Situation two assumed that there were 10-20 hotspots per drum.

The counting geometries that were investigated were distance from the side of the drum [20cm, and 100cm], counting from a single side or from two sides of the drum, fixed or vertically scanning detectors, and stationary or rotating drum.

Table 2 presents the results. For both energies, there are two different standard deviation values. The column labeled “%sdA” is the “normal” or arithmetic standard deviation of the efficiency values, expressed as a percent of the mean efficiency. The column labeled “sdG” is the geometric standard deviation, expressed as a factor of the geometric mean efficiency value. Whereas arithmetic standard deviations are added and subtracted from the mean, geometric standard deviations are multiplied and divided by the mean to yield the upper and lower confidence intervals.

In these analyses, especially at the 60 keV energy, the data are disproportionately distributed on the low energy side of the mean. A skewness evaluation indicates that the geometric standard deviation is

the more proper one to use. As the standard deviation is improved, either by better geometry or higher energy or more hotspots, the skewness decreases and the two standard deviation measures approach each other. Both are presented here for comparison.

distance	motion	hotspots	60 keV		1000 keV	
			%sdA	sdG	%sdA	sdG
20cm	stationary	1-5	256	28.00	81	2.44
20cm	scanning	1-5	300	27.00	93	2.50
100cm	stationary	1-5	184	18.50	60	2.02
20cm	rotate 180deg	1-5	167	6.33	43	1.49
100cm	rotate 180deg	1-5	115	3.94	25	1.28
20cm	rotating	1-5	88	2.80	22	1.24
20cm	scan+rotate	1-5	85	3.24	24	1.27
100cm	rotating	1-5	89	3.30	23	1.26
20cm	stationary	10-20	71	2.10	20	1.23
20cm	scanning	10-20	70	2.31	22	1.25
100cm	stationary	10-20	48	1.73	15	1.17
20cm	rotate 180deg	10-20	46	1.63	11	1.12
100cm	rotate 180deg	10-20	37	1.50	8	1.09
20cm	rotating	10-20	30	1.40	8	1.08
20cm	scan+rotate	10-20	28	1.36	8	1.08
100cm	rotating	10-20	20	1.24	5	1.05
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Several trends can be seen from the data.

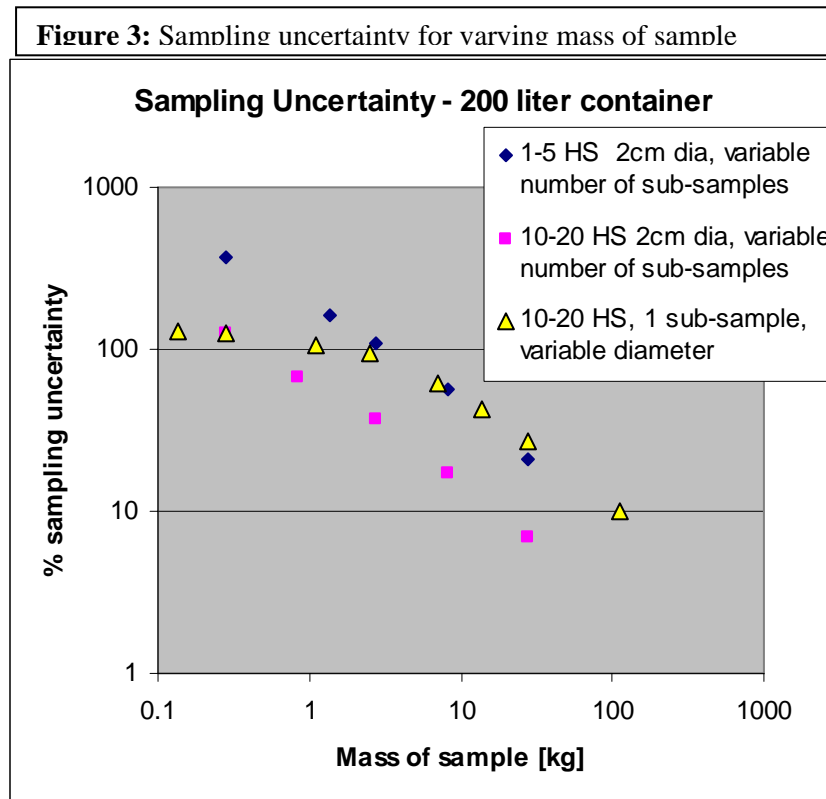
- Low energies have considerably higher standard deviation than high energies
- A detector up close at 20cm to a stationary drum has the highest standard deviation
- Scanning the detector up and down the full drum height doesn't improve the standard deviation very much for this situation where the radioactivity is randomly distributed, but might be useful if there were the potential for the hotspots to settle
- Moving the detector back to 100 cm definitely helps, but also reduces the efficiency a factor of 2 at low energies and 4 at high energies, and therefore will increase the counting statistic component of the total propagated uncertainty; alternatively, the measurement time must be increased a factor of 4-16
- Keeping the detector at 20cm and rotating it 180 degrees half-way through the count is better than a stationary drum and retains the high efficiency
- Rotating the drum 180 degrees half-way through the count with the detector at 100cm is somewhat better than a stationary drum
- Continuously rotating the drum during the count is the best, and it is somewhat better at 100cm than 20cm; it doesn't help very much if scanning is added to rotation but that may be an artifact of this example
- Increasing the number of hotspots dramatically reduces the standard deviation for all geometries and for all energies

A very large component of the efficiency uncertainty estimate is the number of hotspots in the drum. At 60 keV, if there are 1-5 hotspots, the uncertainty is a factor of 28, while if a reasonable assumption can show that there are 10-20 hotspots in the drum, then the uncertainty is only a factor of 2 for the simple and efficient 20cm stationary count, and down to 30-40% with the better geometries. At high energies, even when up close, the uncertainty is a factor of 2-3 for the simple up-close stationary count, reducing down to a 5-10% with the better geometries.

#### 4.2 Non-homogeneous distribution of radioactivity in 200 liter drums – sampling



In this exercise, the same containers with random hotspots used in the previous section were sampled using the IUE sampling function. In the first sampling strategy, top-bottom core samples were “taken” from each container. In one case, the sample diameter was held at 2cm and various numbers of these 2cm samples were combined into a composite for the laboratory analysis. In another case, only 1 sample was taken but the diameter of it was varied. Figure 3 shows those results. It can be seen that



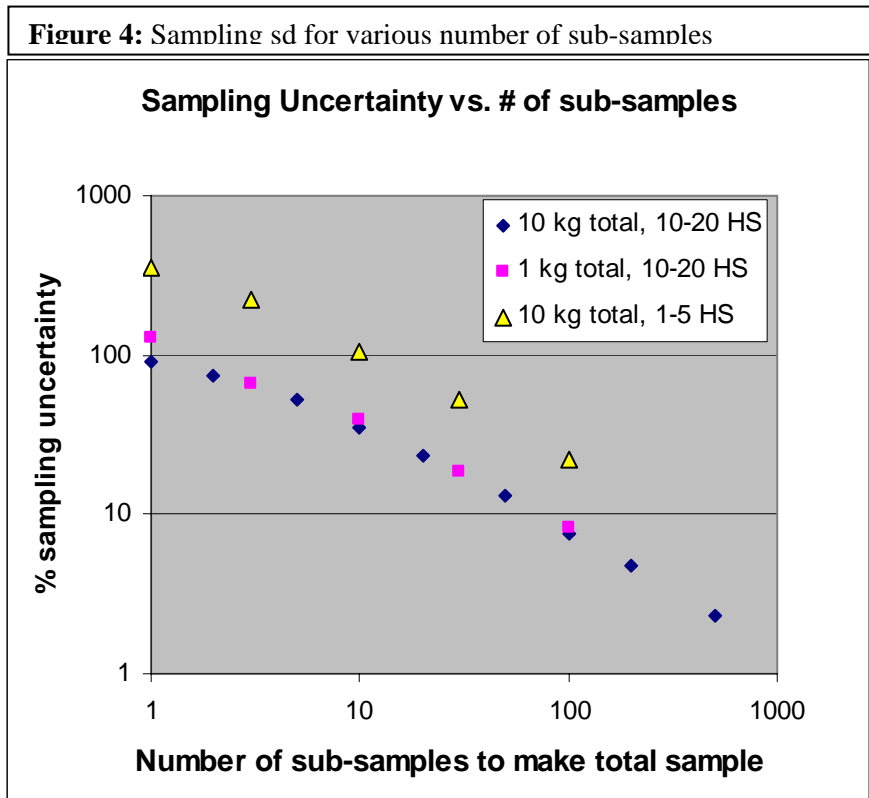
the sample uncertainty is considerably higher than the *in-toto* uncertainty [20-40% is easy to do] until the total sample volume approaches the 10-20 kg range.

The second sampling strategy to be explored held the total mass at 10 kg or 1 kg, and varied the number of sub-samples needed to make up that mass. Figure 4 shows those results. This data shows that even if the sample mass is 10 kg, it must be derived from 10-100 sub-samples to give the same 20-40% uncertainty as the *in-toto* measurement. While these sampling conditions are possible, they are certainly quite unrealistic and rarely performed.

## 5. Conclusion

It has been shown that for rather extreme non-homogeneous distributions, there are several reasonable easy to implement *in-toto* measurement strategies that can reduce the measurement uncertainty down to the 20-40% sd range and lower.

It has also been shown that for these same non-homogeneous distributions, any practical sampling strategy will have a considerably higher uncertainty. Therefore even a perfect laboratory measurement when propagated with the sampling uncertainty will have a total uncertainty higher than the *in-toto* result.



As a commentary – is it really necessary to have a very low standard deviation? No – but what IS required is to accurately present the quality of the result so that the proper decision for that container can be made. For example: if the measurement result for Am-241 at 60 keV was a factor of 100 below the “decision limit” then even the quick and simple 20cm stationary measurement with a measurement uncertainty of a factor of 30 would be adequate to prove that the item is “acceptable”. If most of the containers are like this, then this simple geometry is an economical one to use. If then a few of the containers have results closer to the limit [perhaps within 2sd or a factor of 60], then those few could be recounted in a more precise method – perhaps on a rotating platform at 100cm.

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