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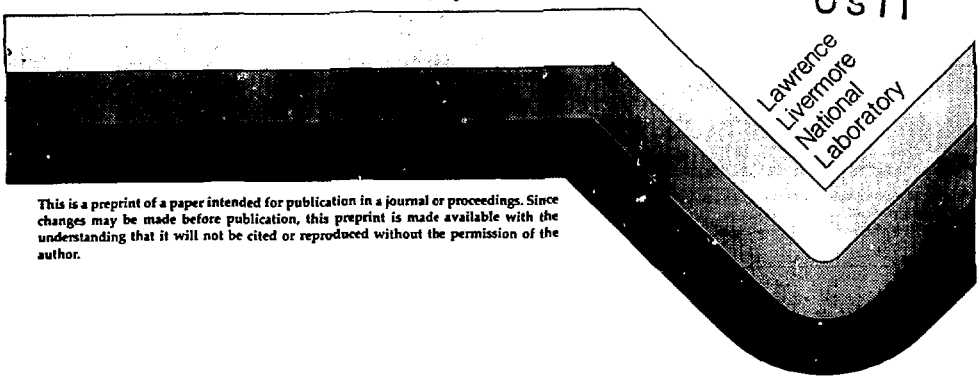

DISCLAIMER

Preliminary Minimum Detectable Limit Measurements in 208-L Drums for Selected Actinide Isotopes in Mock-Waste Matrices

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Abstract

Preliminary minimum detectable levels (MDLs) of selected actinide isotopes have been determined in full-scale, 55-gallon drums filled with a range of mock-waste materials from combustibles ($0.14\ g/cm^3$) to sand ($1.7\ g/cm^3$). Measurements were recorded from 100 to 10,000 seconds with selected actinide sources located in these drums at an edge position, on the center axis of a drum and midway between these two positions. Measurements were also made with a ^{166}Ho source to evaluate the attenuation of these mock-matrix materials as a function of energy.

By knowing where the source activity is located within a drum, our preliminary results show that a simply collimated 90% HPGe detector can differentiate between TRU (>100 nCi/g) and LLW amounts of ^{239}Pu in only 100s of measurement time and with sufficient accuracy in both low and medium density, low Z materials. Other actinides measured so far include ^{235}U, ^{241}Am, and ^{244}Cm. These measurements begin to establish the probable MDLs achievable in the nondestructive assays of real waste drums when using active and passive CT. How future measurements may differ from these preliminary measurements is also discuused.

Introduction

Both in the United States and throughout the World, massive amounts of radioactive waste exists. For example, spent fuel rods from either commercial or university research reactors have activities of several million Curies per cubic meter ($10^6\ Ci/m^3$), and most of these wastes are currently stored near the reactor of origin. Within the U.S. and more specifically within the Department of Energy (DOE) there are four major types of radioactive wastes. High level wastes (HLW) have activities of about $10^3\ Ci/m^3$; transuranic wastes (TRU) have activities of about 14 Ci/m^3; low level wastes (LLW) have activities that range from 0.5 to 6 Ci/m^3; and uranium mill tailings have activities of only 0.01 Ci/m^3. The latter two categories account for almost all of the volume (~99%) of wastes. Both TRU and LLW may contain hazardous materials, and these wastes are known as "mixed." Before 1970, all waste but spent fuel were buried in land trenches and backfilled, and the buried wastes may or may not have been containerized. Since 1970, much of the wastes have been containerized; some has been buried, but most TRU wastes have been shipped to or stored at DOE sites. More information about each of these types of wastes, and especially low level wastes, can be found in reference 1.

The definition of "waste" has evolved over the past two decades. Presently, numerous federal governmental agencies impact the definition. Coupled with state government agencies, these evolving definitions will determine the waste repository guidelines. In this paper we focus on the differentiation between LLW and TRU because of the greater than order of magnitude more cost associated with the disposal of TRU compared to LLW. We are investigating a gamma-ray

based technology that will differentiate between these two waste categories. Currently, TRU is defined as those wastes having in excess of 100 nanoCuries per gram (100 nCi/g = 370 Bq/g) of alpha emitting isotopes with halflives in excess of 20 years. DOE's LLW are those having less than 100 nCi/g of specific activity [1]. Non-DOE LLW (from nuclear medicine, commercial reactors, universities, etc.) are subdivided into Class A, B, C and C+ type wastes that are defined in Chapter 10, Code of Federal Regulations, Part 61 (10 CFR 61).

Some years ago, wastes having an activity of less than 2 nCi/g could be buried in non-regulated land fills. This category of "below regulatory concern" (BRC) wastes has been suspended and has yet to be redefined by the DOE, NRC and EPA. Soon DOE will begin a major deactivation, decommissioning, decontamination and dismantlement (DDD&D) program for its facilities. This will generate considerable amounts of LLW and some TRU wastes. Whatever nondestructive assay (NDA) technologies are used to characterize these wastes, they must be able to differentiate between the various categories and types of waste; and provide sufficiently accurate and quantitative isotopic data to "certify" the proper waste category for disposal.

Each category of waste will be stored or buried in different repositories. TRU wastes are currently planned to be sent to the Waste Isolation Pilot Plant (WIPP) near Carlsbad, New Mexico. In the past, DOE's LLW were shipped to the Nevada Test Site for shallow land burial, but that practice has been suspended. Thus, at the present time all DOE sites must store both their TRU and LLW until appropriate disposal sites are opened. Even then, wastes will have to be transported to these sites; hence, each shipment must satisfy Department of Transportation (DoT) regulations. Taken together, all of the regulations from the various federal agencies combined with regional or state regulations will require that each waste container have a ***certifiable*** and ***quantitative*** measure of most, if not all, radioactive isotopes prior to transportation and subsequent burial. The only cost-effective way to carryout the required characterization of these waste will be to use a *combination* of nondestructive assay (NDA) measurements. It is prohibitively expensive to open every waste container to inspect, assay and certify their contents.

There are a number of techniques used throughout DOE to try to quantify the levels of radioactivity within waste containers. Continuing R&D will be required for a variety of measurement technologies before the "accepted" methods are determined. Here, we report on a specific gamma-ray-based measurement technology that seeks to determine preliminary minimum detectable limits (MDLs) for selected actinide isotopes as a function of measurement time in mock-waste matrices contained in 55-gallon drums. Clearly, for a specific gamma-ray energy, the less the activity, or the greater the attenuation by the wastes, or both, the longer the counting time must be to detect any gamma rays emitted.

Those factors most important for MDLs are the energy of the gamma-ray, the counting time, the waste-matrix attenuation, and the *total* background in the vicinity of a peak of interest. The total background consists of radiation from any shielding materials used, the natural background, and any radioactivity intrinsic to the drum being counted. Clearly, the more intense the background, the higher a source specific activity must be to be detected, and the higher the MDL will be. Shorter counting times result in fewer counting statistics, which limits the MDL. Therefore, MDLs are a function of sample activity, matrix attenuation and count time; and for any NDA system, achieving lower MDLs means using longer counting times, all other factors being equal.

Present vs Future Technology

The current practice within DOE for characterizing nuclear wastes include three nondestructive measurement techniques. First, there is the nondestructive evaluation (NDE) technique of real-time radiography (RTR) that portrays on a TV screen x-ray images of the contents in a waste drum so that nonconforming materials (e.g., free liquids, pressurized containers, etc.) can be identified. Second, there is the nondestructive analysis (NDA) technique of passive and active neutron (PAN) interrogation that seeks to determine the amount of spontaneous (passive) and induced (active) fissionable isotopes present in the wastes. Third,

there is the NDA technique of segmented gamma-ray scanning (SGS) spectrometry that seeks to measure the amount of only ^{235}U or ^{239}Pu detectable in a waste drum. Of these techniques, only the RTR capability is adequate; however, it does not measure any kind of radioactivity within the wastes. Since this paper primarily addresses the gamma-ray measurement capabilities, we do not discuss the RTR or PAN techniques any further, but there is general agreement throughout DOE that improvements are needed in all of the neutron-based interrogation techniques used.

The SGS technique was originally developed to measure the spatially-averaged, passive, gamma-ray intensity of ***one gamma-ray*** from either ^{239}Pu ***or*** ^{235}U in ***homogeneous*** waste matrices. Unfortunately, the use of this technique has been extended to measure heterogeneous wastes; so at best, it provides only a semi-quantitative value for either actinide. Since SGS does not measure the entire spectrum of gamma-ray energies emitted, it cannot provide a complete inventory of all isotopes of interest. WIPP's waste acceptance criteria (WAC) calls for the quantification of thirteen different actinide isotopes [2], while all LLW waste types may include not only the actinides, but also activation and fission products as well. Thus, it is essential to record the entire energy spectrum so that all detectable isotopes can be quantified.

A typical SGS passive measurement consists of from 5 to 15 horizontal slices or segment measurements of 100 to 300 seconds (s) duration for each segment of a revolving drum. For the active measurement, a collimated source, e.g., ^{75}Se or ^{152}Eu, is transmitted through a revolving waste drum and provides a single, integrated-average attenuation value for each drum segment. This value is applied to correct each respective passive gamma-ray intensity segment value for attenuation, and the 5 - 15 corrected values are summed to obtain a measure of total drum activity. The active measurement also requires 100 s or more per segment, and may or may not be taken simultaneously with the passive measurement. Thus, a complete SGS measurement may take 600 to 10,000 s (including all non-measurement times required to move a drum to a new segment). Precisions reported for SGS assays are $\pm$100% for 1 gram (g) of plutonium in 55-gal. drums, $\pm$10% for 10 g, and $\pm$3% for 30 g, respectively [3]. The maximum allowable ^{239}Pu content in a drum differs from site to site, but must be < 200 g to meet WIPP's WAC disposal requirements.

To understand what is required by WIPP WAC in measuring actinide content, Fig. 1 shows a generalized ***semi-log*** plot of actinide isotopic content in grams plotted as a function of total net weight of waste in a 55-gal. (208-*L*) drum. The plot divides into 3 regions. The TRU region lies above the 100 nCi/g threshold, which increases linearly with the weight of a drum. The former BRC threshold of 2 nCi/g is also shown; thus, the region between is defined as LLW. For a net waste weight of 100 kg or 10^5 g, a drum would need to contain only 10^5 x 100 nCi/g or 10 mCi of activity to be at the LLW-TRU threshold. For the actinide ^{239}Pu, a 100 kg 55-gallon drum would need to contain only 160 ***milli***grams of ^{239}Pu to have the drum no longer be LLW, but become TRU wastes. SGS, *as now practiced throughout DOE*, is not capable of making an *accurate* assay for this small amount of Pu. If there were only 3 mg of ^{239}Pu in this drum, then it would formerly have been designated BRC. No current gamma-ray based technique can provide sufficiently accurate quantitative values for this small amount of ^{239}Pu that are *properly corrected for gamma-ray attenuation from heterogeneous wastes* in either the LLW or the TRU region. Thus, at present, most wastes cannot be "certified" as either LLW or TRU.

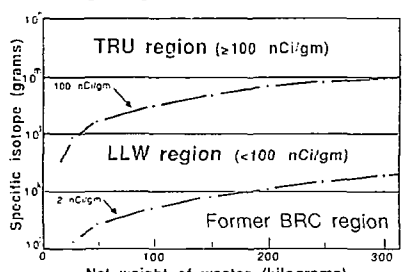

Fig. 1. Semi-log plot of isotopic mass vs. net waste weight in 55-gal. drums. *Three regions are shown. See text.*

Non-DOE LLW wastes, which may contain activation and/or fission product isotopes but little if any transuranic isotopes, are divided into four classes of LLW: A, B, C, and C+. These classes of wastes require a slightly different plot than Fig. 1. However, the same requirements exists for their activity content values; that is, they must be quantitative and ***properly corrected*** for the ***heterogeneity*** of the waste matrix before waste can be "certified" ready for transportation to, and disposal at, an appropriate LLW site.

At LLNL, we are directing our R&D efforts at improving the accuracy and senstivity of gamma-ray based NDA methods. In particular, we are investigating the use of gamma-ray based computed tomography (CT) to better quantify the radioactivity assay values [4]. Transmission or active (A) CT uses a collimated gamma-ray source that emits multiple mono-energetic gamma rays to noninvasively and simultaneously map the attenuation caused by a heterogeneous waste matrix as a function of energy [5] and provide effective atomic numbers (Z) of the waste materials [6]. ACT is done one segment at a time; thus, the entire contents of a drum can be imaged, digitized and displayed in two (one segment) or three dimensions (3-D) (entire drum) [7]. Emission or passive (P) CT records the entire gamma-ray energy spectrum in a similar tomographic manner for the same waste container; hence, PCT can in principle ***localize*** all detectable radioactivities to a specific volume element in the waste matrix [6]. Since the entire spectrum is recorded in the PCT mode, specific radioisotopes can be ***identified*** from their characteristic gamma-ray spectra. By combining source localization results from PCT with the proper energy dependent ACT attenuation map results, we can ***properly correct*** any detected radioactivity for attenuation caused by the waste matrix, regardless of where the source was located or what materials the radiation passed through. In this way, we obtain ***accurate*** and ***quantitative*** activity values for every isotope detected within the wastes. Having accurate activity values provides the necessary data to "certify" wastes so that they can be transported and disposed of *legally* and at the least expense.

Experimental Equipment and Measurements

For our preliminary experimental measurements we have chosen to use a p-type, ORTEC detector. This detector has a 90% relative efficiency (to a 3"x3" NaI(Tl) detector at 1.3 MeV), an 80/1 peak-to-Compton ratio and 2.1-keV FWHM energy resolution at 1.33 MeV. One centimeter in front of this detector we placed a 5-cm-diameter x 10-cm-long lead collimator lined with cadmium and copper to reduce scattered lead x-ray intensity. Signals from the built in preamplifier were routed to an ORTEC 973U ultra-high rate amplifier and then processed by an ORTEC 921 *Spectrum* MASTER©. We used a Dell 386-33 PC for data acquisition with an ORTEC MCA board. Standard bus interface was used for communicating between the PC and the *Spectrum* MASTER©. Most of our measurements were made with the front of the lead colllimator located approximately 3.0-cm from the outside of a drum.

We created three stainless steel, 1.5-mm-wall thickness, 55-gal. (208-*L*) drums as follows. Each drum was divided into three vertical sections; the bottom and top thirds remained empty. In the three middle sections we placed mock-waste materials of (1) very low density and low Z, i.e., combustibles such as paper, booties, lab coats etc.; (2) materials of low density and mixed Z such as broken glass, machine turnings, etc. placed into plastic bags; and (3) materials of medium density and low Z like sand and concrete placed in plastic bags for easy placement and removal. In each middle section three aluminum tubes were fastened to the bottom, each with a 2.7-cm inside diameter and 1.5-mm-wall thickness. These three tubes were positioned on the center axis of the drum, near the drum perimeter, and mid-way between these two, and allowed us to place a source within each matrix type at a selectable height, and in any one of the three radial positions. A circular aluminum turntable, marked every 22.5°, allowed us to rotate and reposition a drum.

In this paper we report on preliminary measurements made for the determination of MDLs for selected actinide isotopes. The isotopes (and their respective masses shown in parentheses) are: ^{235}U (2.6 g), ^{239}Pu (0.4 g), ^{241}Am (2.9 mg), and ^{244}Cm (18.5 mg). The ^{239}Pu source was one of 7 calibration sources with ^{239}Pu abundances ranging from 65% to 94%. We also had a ^{233}U

(0.57 g) and a ^{252}Cf (1.0 μg) source. By placing one of these isotopes in a drum filled with clean, mock wastes we can simulate that same radioactivity in a similar waste type; hence, determine an MDL for each isotope in a selected type of waste. We also used a 75 μCi ^{166}Ho source to evaluate the attenuation within, and transmission through, the various matrix materials as a function of energy. This source is long lived (1200y half-life) with seven major gamma-rays that decrease in intensity from 180 to 950 keV and spaced on average about every 130 keV. Generally for each source, data were recorded for run times of 100 s to 10,000 s, depending on the desired statistics.

For low density, low Z wastes, some gamma-ray sources were too strong. In these cases, a reduced source strength can be "created" by placing a source in the tube located at the mid-radial position and rotating the source away from the detector-collimator axis, (the 0°-180° line). Fewer gamma-rays are recorded depending on the fraction of radiation entering the detector collimator. The amount of source strength reduction is obtained by ratioing data taken with a source placed in the mid-radial tube at say the 90° off-axis position to data taken with the source placed in the central tube, both in an empty drum. When a source was placed in the mid-radial tube at 90°, corrections were applied for the slightly more matrix material and the slight increase in the $1/r^2$ distance.

In real waste drums most passive CT ray paths or projections will show little to no statistically significant data. To acquire PCT scan data rapidly, very little time (1s - 10s) will be spent at each ray path. Even if longer PCT counting times were possible, the number of gamma-ray events recorded would decrease if the source strength were less. Therefore, if a source within a particular segment is weak, very poor counting statistics will result. Thus, we chose to record some gamma-ray spectra using short counting times. Once a scan is completed only PCT ray path spectra associated with a suspected source localization would be summed, and even these may contain only 50 to 200 s of data. Therefore, a gamma-ray spectrum resulting from a short count time and having poor statistics can represent either a longer count time with a weaker source, or a shorter count time with a stronger source. In a future A&P CT system that might use multiple HPGe detectors, the total scan time required for the A and P CT scan modes decreases because *multiple* HPGe detectors are used, but this may not provide any increase in ***counting*** efficiency. The purpose of the multiple HPGe detectors is to acquire multi-path scans simultaneously, and thereby decrease total drum scan time.

Results

The top spectrum in Fig. 2 shows a γ-ray spectrum recorded for 10,000 s with a 94% enriched $^{239}PuO_2$ sample containing 400 mg of total Pu placed in the central drum-axis tube and surrounded by very low density, low Z matrix material. The 129-, 375- and 414-keV lines from ^{239}Pu and the 60- and 125-keV peaks from ^{241}Am are easily identified. The lower spectrum shows net data, i.e., with the background radiation from the room+shield+combustible materials subtracted. These data represent a 55-gallon drum with a net waste weight of only about 30 kg, which translates to an average density of only 0.14 g/cm³.

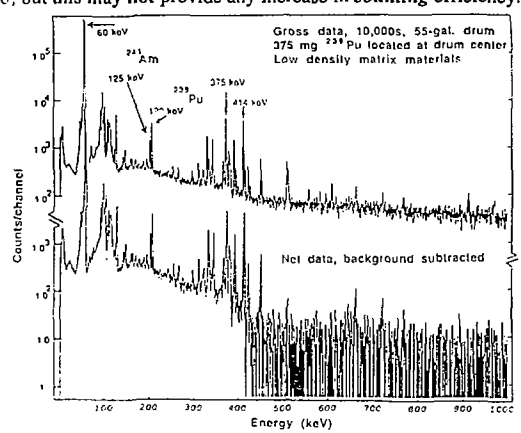

Fig. 2. Gross and net 10 ks spectra of 375 mg of ^{239}Pu in a 55-gal. drum filled with combustible materials.

Figure 3 shows data from the 94% enriched ^{239}Pu sample located in the mid-radial position tube and surrounded by the same combustible matrix materials. Here, the data is accumulated for only 100 s with a drum turned to the 90° position, not 0°. Data taken with this source at this 90° location in an *empty* drum resulted in a counting rate 75x less (corrected for the slight increase in $1/r^2$ distance) than when this source was located in the center axis tube. Thus, Fig.3 represents 100 s of data from an equivalent 5 milligrams of ^{239}Pu. Both the 375- and 414-keV peaks have a net area of 32 counts, hence provide mean counting statistics equal to $32\pm12.5\%$ or 32 ± 4 *counts*, before correction for branching ratios, detector efficiency and matrix attenuation. Thus, the total 2σ error is no smaller than 25% for 5 milligrams of ^{239}Pu in 100 s of counting time in combustible materials. Passive CT tomograms from such data would be reconstructed using narrow energy windows centered on each significant gamma-ray peak of interest and on one or both sides of each peak to obtain background data, which is subtracted to obtain source localization "maps" for each gamma-ray activity in a segment.

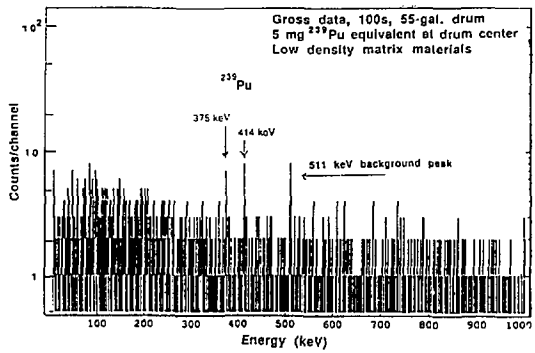

Fig. 3. Gross data for 100 s of 5-mg equivalent of ^{239}Pu in a 55-gal. drum filled with combustible material.

The top 10,000 s spectrum shown in Fig. 4 results from placing the same 94% enriched, ^{239}Pu sample in a mock waste matrix of medium density, low Z sand. The greater attenuation of sand totally absorbs the 129-keV line. Meanwhile, the 375- and 414-keV lines are decreased in intensity by about a factor of 30 relative to their strength in the combustible matrix materials. The net 55-gal. drum mass for sand is 350 kg, which corresponds to an average density of 1.7 g/cm^3. Here also the lower 10,000 s spectrum is obtained by subtracting a spectrum of room + shield + natural sand radioactivity with no ^{239}Pu source. Paper and plastic do not contain any natural radioactivity, but sand does. The greater attenuation of sand is shown by the much fewer ^{239}Pu counts.

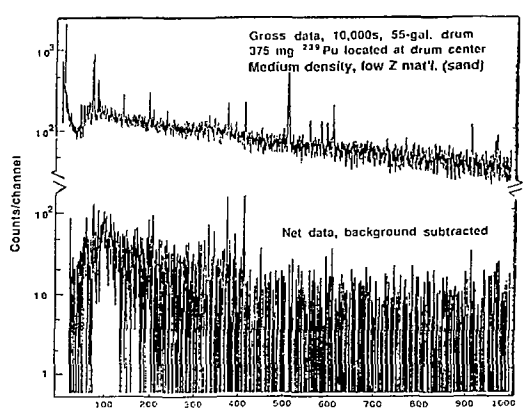

Fig. 4. Gross and net 10 ks spectra of 375mg ^{239}Pu in a 55-gal. drum filled with medium density, low Z material (sand).

Figure 5 shows data for 100 s of counting time for the same 375 mg ^{239}Pu sample located at the drum center in a sand matrix. Here, we record 31 and 43 net counts for the 375- and 414-keV peaks, respectively, or a mean of 37±22% (1σ error) before correction for any gamma-ray attenuation, branching ratios and detector efficiency. Thus, 375 mg of ^{239}Pu can be detected in a 350 kg net weight drum with a 2-sigma error approaching 50%. These data are more representative of real mixed waste containing activity. Note, no background subtraction is made here to the gross spectrum, but PCT tomograms from such data would be obtained by using narrow energy windows centered on each significant gamma-ray peak of concern and on one or both sides to get data for a background, which is subtracted to obtain the net PCT data.

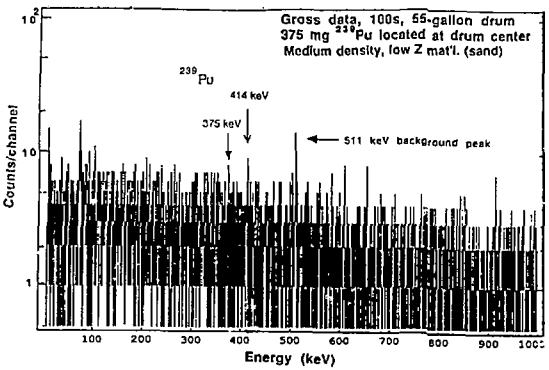

Fig 5. Gross data for 100 s from 375 mg of ^{239}Pu in a 55-gal. drum filled with medium density, low Z material (sand).

Figure 6 shows a plot of the BRC, LLW and TRU regions specifically for the mass of ^{239}Pu contained in a 55-gal. drum vs its net weight. The BRC region is now referred to as "Contamination Below Environmental Concern" (CBEC). On this plot, we can summarize our preliminary MDL results for ^{239}Pu and show where they occur relative to the TRU-LLW threshold. From the results derived from Fig. 3, the MDL of 5 mg of ^{239}Pu in a 30 kg drum had a 2σ error of 25% and is seen in Fig. 6. Similarly, from the results of Fig. 5, the MDL for a 350 kg drum of sand is 370 mg with a 44% 2σ error and is shown in Fig. 6. These data suggest a proposed A&P CT system will be able to quantify LLW amounts of ^{239}Pu in many waste drums to be measured. We also show in Fig. 6 the measurement precisions repor-

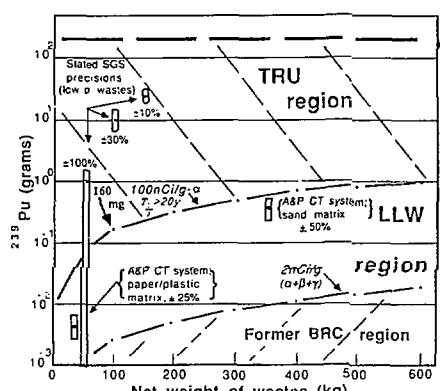

Fig. 6. Plot of mass of ^{239}Pu versus net waste weight. The TRU, LLW and former BRC regions are shown. The MDLs shown at 30 and 350 kg represent only 100 s of measurement time for 5 and 370 mg of ^{239}Pu, respectively, made with a source at the drum center.

ted for the SGS technique assuming *homogeneous* matrix materials having densities less than 0.75 gm/cm^3. The SGS technique is not designed to measure *heterogeneous* wastes with medium to high densities. Our MDL measurements will be repeated once the full-scale A&P CT scanner is completed. More realistic waste matrices will also be employed for representative wastes that span the range shown in Fig. 6.

Table I summarizes our results for ^{239}Pu and the other actinides we have measured to date. In most cases we measured the sources listed in Table I in all three drum locations mentioned above. Thus, ^{241}Am is easily detected at the edge of a drum; but is difficult, if not impossible, to detect in anything but combustible matrices when it is located either half-way toward the drum axis or at the drum axis. In the case of ^{244}Cm, there are two usable gamma-rays one at low energy (153 keV) and one at higher energy (818 keV). So, ^{244}Cm is less sensitive to matrix density than those actinides having gamma-rays below 500 keV. In future measurements, we plan to extend this table to include all thirteen radionuclides required by the WIPP WAC. (These thirteen isotopes are ^{233}U, ^{237}Np, ^{236,8,9,40,1,2}Pu, ^{241,3}Am, ^{242,4}Cm and ^{252}Cf). Some of the MDLs for the isotopes of Pu other than ^{239}Pu will be derivable from measurements already made with the present source. Finally, it must be emphasized that the prototype A&P CT scanner now under construction may employ more collimation than the simple collimator used in these experiments, hence the MDLs may differ from those values shown in Table I. Also as stated earlier they will depend on the amount of intrinsic radioactivity in the waste, the waste matrix density and the total passive scan time for those scan-paths within a segment that contain any gamma-ray radioactivity.

TABLE I. Preliminary Minimum Detectable Limits for Selected Actinides in 55-gal. Drums

Isotope	Gram amount	Energy (keV)	Location in drum	Preliminary MDLs versus matrix of	
				paper/plastic *	sand/cement **
^{239}Pu	0.400	129 375 414	Edge	< 5 mg	~ 10 mg
		375 414	Center	~ 10 mg	~ 400 mg
^{235}U	2.60	185	Edge	< 5.0 mg	~ 5.0 mg
			Center	~ 50.0 mg	~ 1.5 g
^{241}Am	2.9 μ	60	Edge	< 1 ng	~ 1 ng
			Center	> 10 ng	–
^{244}Cm	18.5 m	153	Edge	~ 1 mg	~ 3 mg
		818	Center	< 5 mg	~ 50 mg

* Drums with net waste densities ≤ 0.25 g/cm^3; ** Drums with net waste densities ~ 1.5 - 2.0 g/cm^3

Summary

These preliminary MDL measurements indicate that an active and passive CT drum scanner would be able to differenitate between LLW and TRU levels of ^{239}Pu in low to medium density waste matrix materials of low atomic number and in short duration counting times that would be typically used to obtain passive CT data. We plan to extend these measurements to the remaining 10 actinides required by WIPP's waste acceptance criteria (WAC).

We are currently constructing a full-scale, 55-gallon, A&P CT waste drum scanner that will utilize the 90% HPGe detector used in this work. We plan to measure the existing mock-matrix materials and other waste types frequently encountered such as drums filled with poly bottles

containing glass vials, Raschig rings and stainless steel Vollrath cans, as well as other simulated matrix materials that more fully span the range of equivalent waste densities shown in Fig. 6.

As already mentioned, the A&P CT scanner requires collimation in both modes. The collimation used in the passive mode may be more restrictive than that used to obtain the data in Table I; hence, the emphasis in this paper on "preliminary" MDLs. Once we have chosen that collimation, we will remeasure the MDLs for the actinides shown in Table I along with others necessary for the WIPP WAC. We also plan to begin at that time to measure selected activation and fission products to determine MDLs applicable to Class A, B, C and C+ type LLWs. These future MDLs will also establish the required passive scan times necessary to reach the lower part of the LLW region. Once the prototype A&P CT scanner is operating, we will also measure real wastes in order to establish more realistic MDLs and their associated accuracies for radioactivities that fall within both the TRU and LLW categories.

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