Title:

CONF-980733--

PRELIMINARY REPORT OF THE COMPARISON OF
MULTIPLE NON-DESTRUCTIVE ASSAY
TECHNIQUES ON LANL PLUTONIUM FACILITY
WASTE DRUMS

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Submitted to:

INMM 39th Annual Meeting
Institute of Nuclear Materials Management
60 Revere Drive, Suite 500
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PRELIMINARY REPORT OF THE COMPARISON OF MULTIPLE NON-DESTRUCTIVE ASSAY TECHNIQUES ON LANL PLUTONIUM FACILITY WASTE DRUMS

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Abstract

Prior to disposal, nuclear waste must be accurately characterized to identify and quantify the radioactive content. The DOE Complex faces the daunting task of measuring nuclear material with both a wide range of masses and matrices. Similarly daunting can be the selection of a non-destructive assay (NDA) technique(s) to efficiently perform the quantitative assay over the entire waste population.

In fulfilling its role of a DOE Defense Programs nuclear User Facility/Technology Development Center, the Los Alamos National Laboratory Plutonium Facility recently tested three commercially built and owned, mobile nondestructive assay (NDA) systems with special nuclear materials (SNM). Two independent commercial companies financed the testing of their three mobile NDA systems at the site. Contained within a single trailer is Canberra Industries segmented gamma scanner/waste assay system (SGS/WAS) and neutron waste drum assay system (WDAS). The third system is a BNFL Instruments Inc. (formerly known as Pajarito Scientific Corporation) differential die-away imaging passive/active neutron (IPAN) counter.

As part of this commercial trailer testing program, 25 Plutonium Facility waste drums were selected to be measured on these instruments. These included six different matrices: combustible, glass, HEPA, metal, plastics, and salts. Because this waste is continually generated over time and shipped from the site; only a limited supply of drums are available at any one time. These items are measured in our NDA Analytical Laboratory within the Plutonium Facility. Two primary techniques have been used on these wastes. For low density waste, a segmented gamma scanner is used. For high density waste a neutron coincidence counter is used. In an effort to improve the facility's ability to measure this waste, the Plutonium Facility has taken the approach of measuring each waste item prior to loading into the 55 gal (208 liter) drum. These items are in

cans ranging from 1 quart to 5 gallons. The assay value for each can in a drum is then summed to generate the total drum contents.

In an effort to increase the value of this comparison, additional NDA techniques at LANL were also used to measure these same drums. These are comprised of three tomographic gamma scanners (one mobile unit and two stationary) and one developmental differential die-away system.

Although not certified standards, the authors hope that such a comparison will provide valuable data for those considering these different NDA techniques to measure their waste as well as the developers of the techniques.

Introduction

This is a preliminary report of the activities currently going on at LANL in which facility waste drums are used to compare, contrast and improve nondestructive assay techniques. (The authors expect to complete data collection in the future and present the final report at the 6th Nondestructive Assay Waste Characterization Conference in November 1998.) As stated in the abstract, this work was started under the auspices of an industrial partnership between LANL and two different NDA equipment manufacturers, Canberra Industries and BNFL Instruments Inc. (BII). The number of NDA systems used to analyze some or all of the 25 waste drums are currently at a total of nine different instruments. They are as follows:

- 3 - Tomographic Gamma Scanners (TGS) (one mobile and two stationary - all from LANL)
- 1 - Neutron Coincidence Counter (LANL Plutonium Facility - TNC: NMT-4)
- 1 - Neutron Coincidence Counter (WDAS: Canberra mobile system)
- 1 - Segmented Gamma Scanner (LANL Plutonium Facility - SGS: NMT-4)
- 1 - Segmented Gamma Scanner/Waste Assay System (SGS/WAS: Canberra mobile system)
- 1 - Combined Thermal/Epithermal Neutron System (CTEN: NIS-6 - Differential Die-Away Technique not associated with the Plutonium Facility)
- 1 - Differential die-away Imaging Passive/Active Neutron (IPAN: BNFL Instruments Inc. (BII))

Objectives of the comparison measurements

The design of the plutonium facility (1970s) placed the NDA lab in building areas with high neutron and gamma background levels. With the new emphasis directed toward resolving between low level and transuranic waste, LANL is interested in quantifying any benefit from assay systems located outside the plutonium facility's high background radiation levels. One of the most difficult aspects of NDA measurements are the measurement uncertainties associated with process measurements. This is particularly true for waste streams where calibration and measurement control standards for NDA instrumentation do not match the matrix of the waste being generated. Performing measurements on a wide range of actual facility waste with multiple

NDA systems yields consistency checks or a lack thereof between systems, including those of the NDA laboratory.

The limitation of using real waste for a comparison is that the plutonium content is not known. This leads to further questions, such as: Where is the plutonium distributed within the drum? Are there other radioactive constituents within the drum? Does the density of the waste inside the drum vary significantly? These are valid questions for any unknown waste item. How do these factors affect an NDA system's ability to accurately measure the waste item? If the accuracy of a system is defined by the measurement of standard materials, which are used to calibrate the system, what assurance is provided to the end user that the measurement results of unknowns are accurate. This is a very difficult question to answer. A comparison study is one approach to begin to determine a measure of accuracy.

Since different interferences can affect different NDA techniques, agreement by multiple techniques on the same waste drum is validation of consistency between these techniques. One the other hand, all NDA methods are subject to large errors in some matrices, so when the results from different methods diverge, it is often hard to judge which method is producing the wrong result. An exception to this is comparisons involving TGS assays. TGS images (to the extent they are correct) reveal the three dimensional distributions of radionuclides and the matrix density within the drum. From these images we can determine the degree of heterogeneity of the sample and can qualitatively predict, for example, that a CTEN assay will be high (overcorrected) because the Pu is concentrated near the edge of the drum. It is generally recognized that the TGS is the most accurate method available over the widest range of waste forms [10]. This limited comparison is to provide preliminary instrument performance information on real waste.

Waste Drum Contents

All of the waste drums used in the comparison were selected from the Plutonium Facility inventory. The range of plutonium loading within the drums was limited by the quantity of drums on site at the time of the initial testing. Each of the drums selected contains radioactive waste generated from an active waste stream at the facility. (graphite, combustible, plastic/kimwipes, rubber gloves, HEPA filters, metal, salts, glass). All of the waste items are assayed individually in the NDA laboratory prior to placement within the drum. One of two NDA systems are used on each waste item. For low density waste an SGS is used. For high density waste a neutron coincidence counter is used. The assay value for each waste item in a drum is then summed to generate the total drum contents.

The following are definitions of the drum contents.

The drum containing the graphite waste are generated from the machining processes at the plutonium facility. The density of the graphite is 1.8 g/cm^3 . Based upon density information provided by the TGS assay, the drum appears to be one third full of waste. This waste is inside one or two gallon sliptop cans inside the drum.

The waste stream indicated by combustible contains low density waste (normally less than $.1 \text{ g/cm}^3$). This includes but is not limited to, cellulose, paper, wood, plastics, kimwipes and rubber. The low density waste items are contained within a plastic bagout bag inside the drum.

The other two combustible low density waste drums selected, plastic/kimwipe and rubber gloves, are filled in the same manner as the combustible drum but contain only plastic/kimwipes or rubber gloves. These combustible drums have been labeled with the name of the waste matrix in the drum.

The drum listed as HEPA filters contains the 8 inch diameter by 6 inch tall and/or the 7 inch square by 6 inch tall HEPA filters. Each filter is encased within a stainless steel protective shell (0.108 inch thick). The filter items are in plastic bags inside the drum. Based up on the TGS density analysis the drum appears to be $3/4$ full.

Drums listed under the metal category contain only high density waste items. Normally this includes but is not limited to, stainless steel items associated with plutonium processing activities (e.g. pipe, brackets, etc.). These waste items can be bulky and are presently not compacted at this facility. This may allow large voids to exist inside the waste drum. Normally these items are within plastic bags inside the drum.

The drums containing salts are generated from the Pu metal preparation process. These sodium chloride and magnesium oxide salts are placed in a one gallon metal slip-top can. The metal can walls are ????? thick. These drums are about three fourths full.

The drums that contain glass waste are generated from the plutonium chemical reprocessing activities. This includes, but is not limited to beakers, glove box windows, sample vials, and bottles. The larger glassware items are broken and stored in one-gallon slip-top stainless steel containers. When the one-gallon container fills-up with waste it is bagged-out from the glovebox line, measured in the Plutonium facilities NDA laboratory, and then placed within a drum. Each drum may contain up to nine one-gallon slip top stainless steel cans, each full of glass shards. The density information provided from the TGS analysis shows that each of the drums appear to be $3/4$ full.

Definition of NDA Systems

SGS: NMT-4

This SGS is sited in the plutonium facility and is operated by Nuclear Material Technology (NMT) personnel. It can measure up to 5- gallon containers and is called a can SGS. The system was built by JOMAR in the late 1980s (using SGS technology pioneered at LANL). The segment size of the system is 0.5 inch. Total plutonium within the drum was obtained by summing the individual analysis for each item in the drum.

SGS/WAS: Canberra

This system, built by Canberra, is mounted in a mobile trailer and was tested outside the plutonium facility in Los Alamos.

The three different tomographic gamma scanners (TGS) used in the comparison are from Los Alamos National Laboratory.

CST-7: TGS and NIS-5: TGS

The CST-7 TGS is mounted in a mobile trailer. The mobile system (owned and operated by Chemical Science Technology personnel of LANL (CST)) was brought into the protected area of the Plutonium Facility where the measurements were performed. This system was built by personnel in the Non proliferation and International Security division (NIS-5) at LANL. Both the CST-7 TGS and the NIS-5 TGS use a ~100% efficient detector in conjunction with the NIS-5 developed TGS_ARC software in combination with the KHOROS tomographic package distributed by the University of New Mexico for image reconstruction and display on UNIX systems. NIS-5 TGS images are produced in 1 to 2 hours computation time on two parallel 300-MHZ DEC-Alpha "screamer" systems with the TGS_ARC code.

The system labeled as TGS: NIS-5 is the NIS-5 prototype and was set up inside the plutonium facility for the measurements.

TGS: NIS-6

The NIS-6 tomographic gamma scanner (TGS) is the original prototype TGS developed at Pajarito Site (TA-18) at Los Alamos in 1990 through 1993[1-3]. It has been modified since it was described in ref. [3] to use two parallel horizontal and vertical scanners, which allows stronger transmission sources to be used. A detailed description of the current scanner configuration will appear in another paper [4] presented at this meeting (refer to the sections describing the "normal" high purity germanium detector configuration). The standard TGS scan resolution of 6.1 cm (10 x 10 image voxels per layer) was used, with 30 min for transmission scans and 30 min for emission

scans, for a total of 1 hour per assay. The scan data were collected using the scanner control software WIN_TGS[5] and were analyzed using the TGS_FIT[6] image reconstruction code.

TNC: NMT-4

The passive thermal neutron barrel counter (TNC:NMT-4) is operated by NMT division personnel and is located in the NDA lab within the plutonium facility. The counter was built by JOMAR Systems in 1989. The system has the option for an add-a-source correction which has not been implemented since the low density waste are measured on the lab's SGS.

WDAS: Canberra

IPAN: BII

The BNFL Instruments Inc. Mobile Waste Assay System (IPAN/GEA) provides the complete suite of passive and active neutron, plus passive gamma isotopic NDA measurements in a very compact, single load, single measurement format. The passive and active neutron measurements use the IPAN analysis technique that has been developed over the past several years by BNFL Instruments Inc. The active portion of the IPAN technology originally was developed and patented at LANL in the early 1980's under the general "differential die-away" technique methodology. Among the several significant improvements BNFL Instruments Inc. has added to this LANL technology are improved matrix measurements (ABS, MOD formalism), improved combined passive-active performance (4x higher detection efficiency and 3x improvement in interrogation source utilization), and systematic passive and active neutron imaging (3x improvement in average assay accuracy as compared to original LANL PAN technology).

The measurements reported in this report were taken prior to full implementation of the Gamma Energy Analysis (GEA) portion of the mobile system. Therefore, all plutonium was assumed to be weapons grade and all waste was assumed to contain no interfering neutron sources, such as, ^{235}U , ^{233}U , and ^{244}Cm . The GEA system has now been fully implemented and methods for handling varying grades of Pu and interfering sources have been implemented. These methods have been reviewed and accepted by the CAO NDA audit team.

The BNFL Instruments Inc. Mobile Waste Assay System (IPAN/GEA) has successfully undergone a complete CAO WIPP surveillance and has been certified to measure waste for disposal at the WIPP. The CAO audit team performed a complete review of the BNFL Instruments Inc. IPAN system's technique, procedures, algorithms and calibration. The audit team determined that the system was capable of measuring a broad range of matrix types with plutonium loadings up to 120g. It is one of only four systems currently certified to measure TRU waste for disposal at the WIPP.

CTEN Active Assays.

The NIS-6 combined thermal/epithermal neutron (CTEN)[7] assay system is currently under development at Pajarito Site at Los Alamos. It is an improvement on existing passive-active neutron (PAN) assay systems used at several DOE sites. Because of time constraints, only the active assay results were available for inclusion in this paper. CTEN's unique lump correction and recognition capability, reported on at this meeting in a separate paper [8], was not used in the analyses reported here. Details about the design and function of the CTEN instrument can be found in ref [9].

The Analysis

For the comparison to be fair, the same procedure was followed each time drums were prepared for assay. The only information provided with each drum was the matrix of the waste and the stream average isotopic distribution of the plutonium. This is the same information that is provided to our NDA laboratory when waste items are delivered for analysis.

Sometimes this procedure could not be followed. For example, both the IPAN by BII and the SGS/WAS by Canberra had problems with the original assay approach. (This was the purpose of the testing, to determine weaknesses and fix them) For the IPAN counter, a match must be made between the type of waste matrix and a calibration indicies located in a software library. For the waste matrices measured during these tests there was no direct match. As a result of this problem, BII used the first measurements of the waste drums to generate the necessary calibration indicies. After the software library was updated, some drums were measured again. All of the initial data was reanalyzed [12] to produce the data presented in this preliminary report. Another problem arose during the measurement of large Pu loadings. BII noted significant variability in the quantitative results. An investigation revealed a problem with the multiplicity moment calculations. A more robust shift register algorithm that extends the range has now been implemented.

The Canberra SGS/WAS had measured all of the waste drums before a problem with the assay algorithm was detected. Initially the 129 keV plutonium gamma ray was heavily weighted versus the 414 Kev gamma ray based on its higher activity. Higher density materials in some simulated waste drums significantly attenuated the 129 keV gamma ray, but did not have a corresponding effect on the transmission source. The results shown in this report are the re-analyzed assay results using the 414 Kev gamma ray [13].

An exception to the procedure mentioned above was when the drums were provided to NIS-6 for analysis using the NIS-6 TGS and developmental CTEN instrument. The waste drums were physically transferred to the NIS-6 location (TA-18) and the accountability values (quantity of plutonium in grams) were provided. Drums assayed on the CTEN instrument were used to

determine system weaknesses. One such discovery was that when the system was operated in the passive mode, it was unable to handle the high (alpha, n) rates produced in some of the waste drums [10]. Thus the results presented are only from the active mode of the CTEN instrument.

The NIS-6: TGS was used to generate waste drum images for 24 of the 25 waste drums (one was too heavy). The images (generated by NIS-6 personnel) were provided to NMT-4 personnel. Both a transmission image and emission image were provided. Each image type contained both a layer slice (selected by the user) and a summed side image (similar to radiographs). These images have proven to be valuable for viewing level of drum contents, density within the drum, and location of the plutonium.

The following plots #1 - #9 show the assay result of waste items for comparison. All of the uncertainty values are at the two-sigma level. Where multiple drums of the same matrix were available, the x-axis indicates each drum sequentially starting with the number one. The points following that number represent the different instruments results for that drum. The y-axis indicates grams of plutonium.

Instruments labeled in the legend as SGS: NMT-4 and TNC: NMT-4 are sited in the plutonium facility NDA laboratory. As stated previously, the plotted data from these systems, is the summed value of all the individual measurement results of items within the waste drum. The associated uncertainty value was propagated from adding the individual measurement uncertainty values in quadrature. LANL is preparing these drums for measurement on gamma and neutron based drum instruments. This will help determine any benefits associated with the current LANL can measurements.

The results from the CST-7: TGS and NIS-5: TGS are from one measurement of the waste drum. The two-sigma value is from the predicted error of that measurement. It is important to note that the NIS-5: TGS was in the plutonium facility during the data collection process.

The remaining systems (SGS/WAS: Canberra, TGS: NIS-6, WDAS: Canberra, IPAN: BII and CTEN active: NIS-6) all measured each waste drum five times. The result shown is the average of the five measurements. The uncertainty value shown is the average of the five predicted errors.

The following plots #1 through #3, show measurement results for the waste drums containing small amounts of plutonium. Plutonium loading within each of the drums is near or below the minimum detectable level of the TGSs and SGS (nominally around 500 mg Pu). The WDAS result might have been biased high in plot #1 due to the storage of plutonium materials in close proximity to the system. The most sensitive analysis method for these drums is the CTEN or IPAN active assay. If the moderating and absorbing properties of the waste are properly corrected, the active neutron methods (CTEN and IPAN) are the only methods capable of making

TRU/LLW determination in drums with less than 100 kg of matrix material. The drums shown in plot #1 #2 and #3 were all low weight drums, with net masses ranging from 11.77 kg (TRU cutoff of 19mg of Pu) to 26.53 kg (TRU cutoff of 40 mg of Pu).

The large uncertainty values for the assays of the NIS-5 TGS system in plots 1,2 and 3 are because the system was operated within the confines of the Plutonium facility where significant quantities of plutonium exist (thus effecting the stability and magnitude of the radiation background). This might also account for the possible high bias.

Plot #1 Graphite

Plot #2 BLD plastic/Kim

Plot #3 rubber

The results for the measurement performed on the combustible waste are shown in plot #4. Contained within the drums are paper, wood, rubber, and plastic. TGS images (collected by the NIS-6 TGS system) show the nuclear material in each of the drums to be very close the center of the drum in both the axial and vertical orientation. Assay results among the different methods are in good agreement for drums #1 and #3. The preliminary analysis of Drum # 2 shows a discrepancy that cannot be explained by the TGS images. Further analysis of this drum is being pursued for the final report.

Plot #4 comb

There are three drums that contain plastic/kimwipes and gram quantities of plutonium. The TGS image of the drums at the higher plutonium loadings (#2 and #3) shows most of the plutonium near the edge of the drum. For all of the drums the different NDA technique results are closely grouped (excluding CTEN results) as shown in plot #5. As would be expected the SGS/WAS result is in good agreement with the TGS analysis for these low density drums ($<0.1 \text{ g/cm}^3$). A possible explanation for the high and low biased results of the CTEN instrument for drum #2 and #3 are the result of the heterogeneity effect in which the SNM is concentrated in one place where the matrix interference is more or less severe than the drum averaged value. The result is that the analysis overcorrected in drum #2 and under corrected in drum #3.

Plot #5 plas/kim

The comparison which reflects the drum containing HEPA filters is shown in plot #6. The TGS images show this drum to be over three quarters full. The majority of the plutonium appears to be located in one spot near the bottom of the drum. The TGSs and DDT based systems show good agreement. The WDAS shows what might be a slight high bias which again might be due to close storage of Pu. The overall variation are reasonable when considering the quantity of plutonium within the drum. Both the NMT-4 TNC and the NIS-5 TGS instrument are located in a high background area. Thus both systems have a larger sigma value than their respective counterpart.

Plot #6

With the exception of CTEN(active mode only), the effect of the drums containing metal waste and the increased Pu loading in each appears to define an expected trend between passive neutron counting and gamma based TGS systems (possibly an underestimation of the plutonium). This effect can be seen in Plot # 7. TGS images of these drums show a very large variation of size and location of plutonium "hot spots" within the drums.

Plot #7

The measurement results of the drums containing salts show a significant scatter among the different NDA techniques. The reason for this scatter may be attributed to the plutonium lumps normally found in this waste stream. The TGS matrix density and Pu image for drum # 2 in plot #8 are shown side by side in Figure 1. What we observe in the density image is that the drum is about three fourths full with a semi uniform matrix. Assuming a uniform matrix and knowing the net weight of the drum, the estimated matrix density is $.75 \text{ g/cm}^3$. The limit for high accuracy in a TGS assay is about $.5 \text{ g/cm}^3$ local density (not average density) [10]. The TGS Pu image

reveals the concentration of plutonium near the surface of the matrix, where it is only half shielded by the matrix. This type of heterogenous effect is difficult to predict (without TGS analysis) or correct for, as seen in plot #8. Further analysis is planned for these drums.

Plot #8

Figure 1

The drums of glass waste, like all the drums, were assumed to contain only weapon grade plutonium. All of the assays were performed with this assumption. Drum number two in this set of measurements actually contained a small amount of curium. The effect of curium (~ 1 mg of $^{244}\text{Cm} = \sim 200\text{g}$ Pu total in weapon grade plutonium) can be seen in the assay result for each of the passive neutron counters as shown in plot #9. The IPAN-BII system did both the active and passive neutron count on all drums. In the IPAN counter, for any passive assay result of greater than ten grams the algorithm automatically reports the passive result and not the active result. For the drum with curium this was the case. The passive result was at 35 grams plutonium. This problem with curium is an example of the susceptibility of passive neutron counters to produce erroneous results when exposed to unidentified neutron interfering radioactive constituents. A similar problem exists with passive neutron counters if the isotopic distribution unknowingly varies from item to item. The TGSs analysis and active CTEN results flagged this drum as a problem.

This occurrence may be an example of a larger DOE complex problem. Drums of waste provided to Canberra for NDA, at another DOE facility, were said to contain weapon grade plutonium as the only radioactive constituent. When the passive neutron counter results showed kilo-gram quantities of plutonium. An investigation followed and curium was again to blame. A possible solution to this problem is an isotopic measurement for screening waste or a combined gamma-neutron analysis to compare assay results for flagging problem waste items.

Plot #9

Predicted Random Error versus Observed

Most NDA instrument manufacturers provide an estimate of the precision error (random error) as part of the assay result. This is often referred to as the "machine uncertainty". This prediction is most commonly based on an algorithm utilizing counting statistics. One exception is IPAN which BII has stated estimates total error. In an attempt to validate the random error prediction

algorithms, five repeat measurements, without moving the drum, were made. Since the drums were not moved and replaced between runs, this represents an ideal case. No variability due to the drum positioning was captured. In addition, time constrained us to five repeat runs per drum. These statistics can certainly be improved upon. At a minimum one would expect these predictions to be the same or larger than the observed measured random error from the five repeat runs using the standard deviation calculation. So far we have collected 93 sets, of the five repeat runs (a total of 465 measurements), which include measurements from both NIS-6 instruments (TGS and CTEN), both Canberra instruments (SGS/WAS and WDAS), and the BII's IPAN. Of those 93, 47 sets or roughly 50 percent have a predicted random error that is 10% larger than the observed random error. An attempt to correlate matrix or plutonium gram loading to the uncertainty proved futile. These results are still under analysis.

Conclusion

Although the plutonium content within a waste drum is not known, this type of comparison did provide an indication that all NDA systems performed similarly on most of the waste forms tested. In addition, those employing the same fundamental approach, that is, gamma and neutron based system also showed consistency within their approach. Most importantly, these preliminary comparisons on real waste served to uncover NDA system weaknesses which might not have come to light using only standards and simulated matrices.

Future Work

The LANL plutonium facility assay values shown in the comparison data consist of a summed value for the individual assay results for each of the items in the waste drum. To increase the benefit of the comparison the drums will be brought back into the plutonium facility and measured five times on both the gamma and neutron based drum counters. This data will be presented in the final report to be presented in the fall of 1998 at the 6th Nondestructive Assay Waste Characterization Conference.

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Below Limit of Detection - GRAPHITE DRUM COMPARISON

DRUM

Ch-GRAPHIT-1

Below Limit of Detection - Plas/Kimwipe DRUM COMPARISON

Below Limit of Detection - RUBBER DRUM COMPARISON

Ch-RUB-3

COMBUSTIBLE DRUM COMPARISON

PLASTIC/KIMWIPE DRUM COMPARISON

HEPA DRUM COMPARISON

DRUM

Ch-HEPA-6

METAL DRUM COMPARISON

SALT DRUM COMPARISON

Ch-SALT-8

GLASS DRUM COMPARISON