

# DOES THIS MATERIAL CONTAIN DOE-ADDED RADIOACTIVITY? RECOMMENDATIONS OF THE DOE RADWG ANALYTICAL LABORATORY COMMITTEE\*

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## ABSTRACT

In 1991, responding to press reports that hazardous waste slightly contaminated with radioactivity had been shipped from a DOE facility to a hazardous waste disposal company, the Department of Energy halted all DOE and contractor hazardous waste shipments. DOE review of waste disposal criteria and methods across the DOE complex determined that the management and operating contractors were using inconsistent criteria for declaring waste radioactive or non-radioactive. To eliminate this problem, DOE directed its contractors to develop a set of criteria to certify that shipped waste would contain "no DOE-added radioactivity at a statistically defined confidence interval." This certification could be based on process knowledge, i.e., if a waste package had never had any possibility of contamination, or it could be based on "reasonable" application of radiological analyses. Contractors began developing these criteria.

## INTRODUCTION

### Formation of the Research and Development Working Group (RADWG)

It soon became apparent to the smaller research and development laboratories that the development of these criteria was a difficult task, especially when some sites might have an entire Environment, Safety, and Health organization of only a few people. Concerns were also being raised about how to proactively maintain consistency and technical merit in the various institutions working on these criteria. Through the coordinating efforts of the DOE Chicago Field Office, responsible parties at the R&D labs came together in a grass-roots effort to establish a working group to share information, pool resources, and project a larger voice in DOE Headquarters considerations of Moratorium issues. Thus formed, the RADWG tasked itself to examine all issues involved with the Moratorium. These span the range from waste generator training through waste analysis to waste disposal.

Committees were formed to study each of the areas of concern identified, and were tasked with developing consensus standards that all the R&D labs could adapt for their own operations. These committees, being drawn from across the DOE R&D complex, provide a consistent set of standards that the R&D sites could employ.

This paper presents a summary of the Analytical Laboratory Committee's recommendations.

### CHARGE TO THE COMMITTEE

The Analytical Chemistry Laboratory (AL) Committee of the RADWG was charged to develop a set of analytical guidelines for the determination of radionuclides in waste materials. This guidance is intended to assist Department of Energy research and development sites to address the DOE Waste Shipping Moratorium performance objective. Specifically, the performance objective allows the use of reasonable

analytical methods in waste analyses. The guidelines developed by the AL committee are to provide a set of methods that

- are developed by consensus across many R&D facilities,
- are technically reviewed by the members of the committee, and
- represent "reasonable" but not "heroic" use of current technology\*\*

### COMPOSITION OF THE COMMITTEE

The AL committee has members from many of the DOE R&D sites, and represents a broad spectrum of respected professionals. Members of the AL committee are given below. The committee is co-chaired by Richard Pietrzak of Brookhaven National Laboratory and Howard Hall of Lawrence Livermore National Laboratory.

- Howard L. Hall (Co-Chair), Lawrence Livermore National Laboratory
- Richard Pietrzak (Co-Chair), Brookhaven National Laboratory
- Mary L. Adamic, Argonne National Laboratory (West)
- Patrick Beaulieu, Los Alamos National Laboratory
- Daryl Bickbichler, Princeton Plasma Physics Laboratory
- Bob Conzemius, Ames Laboratory
- Vernon R. Cupps, II, Fermi National Accelerator Laboratory
- Angela Davi, Lawrence Berkeley Laboratory
- Patrick Epperson, Lawrence Livermore National Laboratory

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\*\* In order to avoid any questions of conflict of interest or vendor favoritism, we will describe commercial instrumentation in this report only in general terms and specific performance requirements. No vendors will be mentioned by name or product. Readers are referred to the various guides to laboratory instruments for vendor information.

- Sandra Fadell, Pacific Northwest Laboratory
- David Green, Argonne National Laboratory (East)
- Bruce Killand, Pacific Northwest Laboratory
- William Lee, MAC Technical Services (DOE Chicago Operations Office)
- Ron Leuenberger, Princeton Plasma Physics Laboratory
- Peter C. Lindahl, Argonne National Laboratory (East)
- Usha I. Narayanan, New Brunswick Laboratory
- G. Price Russ, Lawrence Livermore National Laboratory
- Roger Sit, Stanford Linear Accelerator Center
- Tim Snider, Battelle Columbus Laboratories
- Christine T. Snyder, Argonne National Laboratory (East)
- Henry Tran, Stanford Linear Accelerator Center

### RECOMMENDATIONS

Following are the specific recommendations of the AL committee. It is not intended that these recommendations be prescriptive, rather the committee intends that this report provide guidance on general methods and approaches to waste analyses, which, if followed, form the basis for defensible laboratory analyses. Individual sites may implement all or some of the methods recommended, as appropriate for their facility. Indeed, methods that the AL committee has not recommended may be used, although the burden of defending those clearly falls upon the user. By working within the framework of the committee's recommendations, a level of consistency can be achieved at disparate sites.

To assist those who desire actual working procedures, we include example procedures as attachments to the formal report. The reader is cautioned, however, that specific procedures should be reviewed against the capabilities and needs of their facility. For example, if facilities in which to safely handle perchloric acid are not available, one should use another method or adapt the given procedure. The committee strongly recommends that a knowledgeable and qualified\* chemist (or preferably, a radiochemist) be involved in implementing the methods given in this report.

Another consideration in applying these methods is the need for representative samples. Discussion of sampling techniques is beyond the scope of this document, but the AL committee recommends representative sampling. Documents such as DOE/EM-0089T, Rev. 1, provide guidance on sampling methodology.

Finally, the committee strongly recommends that sites adapt, implement, and use these methods with a vigorous *commitment* to personnel safety. Like most chemical reactions, the ones used in these methods can pose a risk of serious injury if they are misused. Hazards involved in these analyses, particularly wet ashing operations, cannot and must not be treated cavalierly. With proper controls, the risks involved in

performing these analyses are acceptable and manageable, but they cannot be ignored.

### Overall Philosophy

In developing these recommendations, it has become apparent that three basic points are critical to successfully addressing the Moratorium in the analytical laboratory setting. These can be summarized as:

- "know thy waste,"
- measure cost-effectively,
- a radioactive sample does not always mean radioactive waste.

"Knowing thy waste" means that the analytical lab cannot operate without a good understanding and close inclusion in the entire waste generation and disposal process. Just as generators should have knowledge of their particular process, the analytical laboratories should have a more global knowledge of their site's operations, waste streams, and likely radioactive contaminants. Having this knowledge allows the laboratory to target its resources on the real problem, rather than wasting them on unneeded analyses. As an example, a site that generates only activated waste would be better served by concentrating on tritium and  $\gamma$  measurements, rather than less useful gross  $\alpha$  measurements. A facility that handles Pu would not necessarily have the same emphasis. In no way does this relieve the generators of their responsibility to maintain process knowledge. Ideally, the generator should communicate with the laboratory to ensure that the needs of both parties are met.

To measure cost-effectively means to gain as much information per analysis as possible. To this end, we have recommended methods that address a range of radionuclides wherever possible.  $\gamma$  spectroscopy is an excellent example of such a method.

It should be noted that some samples will be measured by the analytical laboratory as radioactive, yet the waste has not had radioactivity added by DOE operations. Soil is perhaps the best example of this, as soil contains a small but easily measured amount of primordial radionuclides such as  $^{40}\text{K}$ , the uranium series, and the thorium series. Furthermore, the amounts of these radionuclides vary with the local geology, so site-to-site (and perhaps even within a site) comparisons are complex. Clearly, this material was there before the DOE, and should not be mistakenly treated as radioactive waste.

A second possibility is that, should a risk-based *de minimus* level be accepted by the DOE, samples may have measurable radioactivity that is of negligible risk and hence treated as nonradioactive. If such a standard is approved by DOE, the recommendations of this report could be readily adapted to accommodate predetermined release criteria.

### Screening Methods

Screening samples for radioactivity within the analytical laboratory setting may be required for sample transport decisions (to meet DOT regulations and recipient licensing restrictions), for proper handling within the analytical laboratory, or for maintaining personnel health and safety.

\* By "qualified," the AL means the chemist has appropriate academic training and/or experience, and is approved by his or her facility's management structure to execute such an assignment.

However, screening techniques and methods *should not be used as a substitute for more stringent but more generally accepted analytical methods* used to characterize radioactivity in waste streams.

Two methods of screening samples are given below. Both are acceptable, although the use of  $\gamma$  spectroscopy alone may be acceptable for certain classes of samples (e.g., activated metals).

### Method 1

- Liquids:
 

Sample size:	1 ml
Preparation:	Direct mixing with liquid scintillation counting (LSC) cocktail
Counting Technique:	LSC using a fully open analysis window on the LSC counter
Counting Time:	10 minutes
Estimated MDA:	*Varies with instrumentation, radionuclides, and matrix
- Solids:
 

Sample size:	1 g
Preparation:	Weigh out material onto a 2" counting planchette
Counting Technique:	Gas proportional counting using windowed detector
Counting Time:	5 minutes
Estimated MDA:	*Varies with instrumentation, radionuclides, and matrix

### Method 2

- Liquids:
 

Sample size:	1 ml
Preparation:	Direct mixing with liquid scintillation counting (LSC) cocktail
Counting Technique:	LSC using a fully open analysis window on the LSC counter
Counting Time:	10 minutes
Estimated MDA:	*Varies with instrumentation, radionuclides, and matrix
- Solids and liquids:
 

Sample size:	25 ml or 25 g
Preparation:	None

Counting Technique:	$\gamma$ spectroscopy using HPGc or similar high-resolution $\gamma$ detector
Counting Time:	10 minutes
Estimated MDA:	Will vary for different nuclides.

### Analytical Methods

Methods listed in these sections fulfill the intent of the AL committee for cost-effective use in making disposal decisions. Except in the case of tritium, the methods are not based on isotope specific separations. Analyses requiring radiochemical isolation of individual elements are very expensive and slow, thus impractical to this purpose. It is preferable to obtain as much information as possible in a single analysis to minimize cost; therefore, the AL committee recommends gross measurements be used where practical. As an example, if a gross  $\beta$  measurement indicates total  $\beta$  activity is less than 1 pCi/g, then clearly  $^{90}\text{Sr}$ ,  $^{90}\text{Y}$ , and  $^{89}\text{Sr}$  are less than 1 pCi/g.

The reader is cautioned that the following procedures do have limitations, and these should be carefully considered in adapting these recommendations to a specific site. Specific procedural limitations are discussed in the appropriate subsections. An overall limitation of any analytical effort is potential error inherent in sampling a non-homogeneous waste form. As an example, shielding blocks and accelerator magnets are waste streams that may require significant effort in sampling to obtain a sample that is representative.

Another consideration is the appropriateness of the statistical test (MDA) that the AL committee recommends. The committee recognizes that some others prefer the  $L_C$  criterion, where  $L_C = 2.33\sigma_B$  at the 95% confidence level. However, it is critical to note that  $L_C$ , to be properly applied, must include a full consideration of all the components that make up  $\sigma_B$ . It is simply wrong to assume that the dominant portion of  $\sigma_B$  is the statistical counting error -- random errors in various chemical manipulations of the sample, not to mention the sampling errors, must be included. These errors are often comparable to or greater than the counting error. While the same is true of calculations of the MDA, the committee believed that the conservative approach of neglecting other errors was acceptable. The committee recommends against using  $L_C$  without considering the other errors as being unnecessarily conservative and unreasonable.

### MEASUREMENT OF GROSS $\alpha$ AND GROSS $\beta$ ACTIVITIES

#### Instrumentation/Sample Preparation

Recommended instrumentation for gross  $\alpha$  and gross  $\beta$  measurements is a 2-inch thin-windowed (or windowless) flowing gas proportional counter with adequate shielding and materials to achieve stable backgrounds that should be no more than 0.5 cpm  $\alpha$  and 2.5 cpm  $\beta$ . Throughput can be increased, and overall cost decreased if the counter is able to

\* In estimating minimum detectable activity (MDA) values, the only terms considered for our calculations are the detector backgrounds, counting times, and sample size. Conversion of the MDA to activity units requires assumptions on efficiency and other factors. Where the assumptions are critical, the assumed value will be noted. The following equation has been used for the calculations:

$$MDA = \frac{3 + 4.65\sqrt{RB}}{tV} = \frac{3 + 4.65\sqrt{\sigma_B}}{tV}$$

where RB is the background count rate, t is the counting time, and V is the sample size.

count multiple samples automatically, preferably with an automated sample changer. In this case, the counter should operate under computer control, and allow electronic acquisition of its data. Additional time and cost savings are realized if the counter is capable of counting  $\alpha$  and  $\beta$  particles simultaneously.

Gross  $\alpha$  and gross  $\beta$  determinations by proportional counting are generally satisfactory for determining the actual activity in a sample, but there are limitations that require facility-specific consideration in using the methods given. In all cases, the methods require drying a sample on a planchette followed by counting. In so doing, volatile radionuclides can be lost. In particular, halides,  $^{14}\text{C}$ ,  $^{32}\text{P}$ ,  $^{35}\text{S}$ , and high-volatility metals can be lost. A second limitation is that the counting efficiency of a proportional counter is a function of the incident particle energy, especially for  $\beta$  particles. Radionuclides that emit  $\beta$  particles significantly different in energy from the calibration source (typically  $^{90}\text{Sr}/^{90}\text{Y}$  for  $\beta$  and  $^{239}\text{Pu}$  for  $\alpha$ ) will not be quantified as well as those that are closer in energy. Finally, sample preparation is the most important step in reaching the given MDA's. After all, quantitation of alpha emitters in an oil by proportional counting is virtually impossible without destroying the organic matrix. Again, the AL committee stresses that it is necessary to understand the facility's waste streams to evaluate the impact of these limitations.

When in use, the counter should be calibrated to correct for sample self-absorption over the range of 0-100 mg of mass on the planchette. Samples should only be counted within this range. This can be achieved by adjusting aliquot sizes.

Recent developments of gross  $\alpha$  and  $\beta$  counting by time-resolved LSC may become an excellent method for determination of radioactivity in waste samples without complex preparation steps. However, this technology is fairly new, and this committee was not able to prove its performance on waste media yet.

Preparation of waste sample for analysis using a proportional counter requires generating a near-massless sample on the counting planchette. This requires wet-ashing the sample with strong mineral acids and oxidizing agents to destroy any organic material. This digestion can be done by standard manual techniques or using an automated open-vessel microwave digester capable of handling large sample volumes. Open vessel digestion is preferred over the more common closed vessel method due to the capability of handling larger samples and the reduced risk of a pressure excursion compromising a sample. In either case, a laboratory with adequate hood space and safety systems for handling fuming acids is required.

#### "Aqueous" Materials

Aqueous samples are predominantly water-based, but are often found to contain traces of organic material. To safely digest an aqueous sample, it should be vigorously boiled to eliminate any volatile organics that could explode during digesting before the addition of any oxidizing agents. Certain media are resistant to wet-ashing, and will require drastic reductions in sample volume to yield counting samples of less than 100 mg final mass (inorganic phosphates are a good example of this).

Sample size:	50 ml
Preparation:	Wet ashing followed by evaporation onto a planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA :	0.01 pCi/ml $\alpha$ , 0.03 pCi/ml $\beta$

#### Acid/Base Solutions

Acid or base samples may contain traces of organic material. To safely digest an aqueous sample, it should be vigorously boiled to eliminate any volatile organics that could explode during digestion before the addition of any oxidizing agents. Bases should be carefully brought to acidic conditions before ashing. Certain media are resistant to wet-ashing, and will require drastic reductions in sample volume to yield counting samples of less than 100 mg final mass (phosphoric acid is a good example of this). Analysis of samples of concentrated acids requires caution as well -- and in particular, haloacids such as HCl can damage the counting planchette if the chloride is not completely driven away before evaporation. Extreme caution is required in handling strong bases, particularly upon the addition of strong acids.

Sample size:	25 ml
Preparation:	Wet ashing followed by evaporation onto a planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA:	0.02 pCi/ml $\alpha$ , 0.06 pCi/ml $\beta$

#### Organic Solvents

Organic solvents, for our purposes, are organic liquids that can be evaporated between room temperature and 100 degrees Celsius. Occasionally these materials leave high molecular-weight residues that remain "wet" or exceed 100 mg upon evaporation. If this occurs, the samples can be slowly ashed in a muffle furnace to destroy the residue, or the sample can be treated as an oil.

Evaporation of solvents should be separated from wet-ashing operations (which use perchloric acid) as much as possible. Under no circumstances should they be performed in the same fume hood, due to the potentially explosive mixture of condensates that could form in the hood ducts.

Sample size:	5 ml
Preparation:	Direct evaporation on a planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA:	0.1 pCi/ml $\alpha$ , 0.3 pCi/ml $\beta$

#### Oil/Viscous Organics/Organic Solids

Oils and similar materials require the most extensive wet-ashing treatment to destroy the organic matrix. Silicon oils may also require treatment with hydrofluoric acid to destroy the silica residue. Any HF operations should be performed in Teflon or platinum labware. Ashing using a muffle furnace is also acceptable.

Any samples in this class should be heated to about 100 degrees Celsius for at least an hour to drive off any solvents

\* Assuming an efficiency for  $\alpha$  and  $\beta$  detection of 30% for the proportional counter

prior to introducing strong oxidizing acids. Extreme care should be taken in performing the wet ashing procedures on these materials.

Sample size:	1 ml
Preparation:	Wet ashing followed by evaporation on planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA:	0.5 pCi/ml $\alpha$ , 1.5 pCi/ml $\beta$

#### Soils/Rocky Debris

Soil and rocky debris (such as concrete) can be reasonably well analyzed by acid leaching. In this process, a representative sample is ground to ensure a large surface area, mixed with acid, and agitated for 4-24 hours. The solids can be filtered away from the liquid, and an aliquot yielding less than 100 mg final mass can then be evaporated on a planchette. Ultrasonic baths provide a superb agitation source for this method.

Sample size:	1 g
Preparation:	Leaching followed by evaporation on planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA:	0.5 pCi/g $\alpha$ , 1.5 pCi/g $\beta$

#### Inorganic Solids

Inorganic solids include various salts, metals, and alloys. These are best analyzed by total dissolution followed by evaporation of an aliquot containing less than 100 mg of residual mass. As a result, the effective sample size is approximately 100 mg. Generally, dissolution of a gram and taking a 1:10 aliquot is sufficient to yield a good source.

Sample size:	0.1 g
Preparation:	Dissolution followed by evaporation on planchette
Counting Technique:	Proportional counting
Counting Time:	100 minutes
Estimated MDA:	5 pCi/g $\alpha$ , 15 pCi/g $\beta$

### MEASUREMENT OF TRITIUM

#### Instrumentation/Sample Preparation

Recommended instrumentation for the measurement of tritium is a low-background liquid scintillation counter (LSC). The instrument should have an automated sample changer, external source quench correction hardware and software, provisions for correcting for chemiluminescence, and available computer control. Instrument background in the tritium region should be stable and no more than 20 cpm in modern units, and is generally less than 10 cpm in "low-level" instruments.

Certain classes of materials are recommended to be combusted before LSC analysis. Small numbers of samples can be combusted using oxygen bombs. Larger throughput of samples can be achieved using automated systems.

#### "Aqueous" Materials and Acid/Base Solutions

These materials are fairly easily measured for tritium contamination by distilling the water away from any salts or solids in the sample. Ideally, the distillation should be done with basic permanganate (to hold back halides and destroy organics) and the counting sample taken from the middle of the distilled volume. However, simple distillation is usually adequate. The more complex distillation should be reserved for difficult samples.

Sample size:	5 ml
Preparation:	Distillation (simple)
Counting Technique:	LSC
Counting Time:	50 minutes
Estimated MDA:	1 pCi/ml*

#### Organic Solvents/Oils/Viscous Organics/Organic Solids

Samples in this category can occasionally be dissolved directly in scintillation cocktail to yield a satisfactory counting sample. However, many samples of this type are heavily chemiluminescent and require combustion to carbon dioxide and water before counting. If an automated combustion system is available to the lab, it should be used on all samples in this category. If the lab has only the manual oxygen bomb capability, it may be preferable to attempt to count the sample without combustion first, to avoid the labor-intensive bomb preparation. The sample sizes are the same for both methods.

Sample size:	0.1 ml (or gram, for solids)
Preparation:	Direct counting, or combustion
Counting Technique:	LSC
Counting Time:	50 minutes
Estimated MDA:	20 pCi/ml

#### Soils/Inorganic Solids

Non-combustible solids are best assayed for tritium by extraction with water followed by distillation. In this technique, the solid material is ground (if necessary) and agitated with water for several hours. This allows the tritium to exchange with the protons in the water, yielding HTO. The HTO is then collected by distillation and counted.

Sample size:	1 g
Preparation:	Extraction, then distillation
Counting Technique:	LSC
Counting Time:	50 minutes
Estimated MDA:	2 pCi/g

### MEASUREMENT BY $\gamma$ SPECTROSCOPY

#### Instrumentation

Identification and measurement of  $\gamma$ -emitting radionuclides by  $\gamma$ -ray spectroscopy are very powerful techniques. They are essentially non-destructive, only requiring the sample to be transformed into a geometry for which the system is calibrated. Software is available to find, identify, and integrate peaks in the  $\gamma$  spectrum, making data analysis fairly simple. Corrections for matrix effects should not be neglected, although in many cases they are small.

The instrumentation recommended for  $\gamma$  spectroscopy is a high purity germanium (HPGe) or lithium-drifted

\* Assuming an efficiency for tritium of 30%.

germanium (GeLi) diode  $\gamma$  detector assembly placed in a region surrounded by a thick virgin lead shield. For special applications where high resolution is not essential, a thallium-doped sodium iodide [NaI(Tl)] detector may be used. These detectors can be controlled in a variety of ways: from a unit as simple as a PC-based card to complex, workstation based data-acquisition packages. The detector should be chosen to yield as high an efficiency as possible while retaining sufficient energy resolution to distinguish all potential radionuclides in a given waste sample.

### Liquid Samples

Liquid samples are easily measured by  $\gamma$  spectroscopy, since the liquids can be poured into a container of calibrated geometry. That container can then be immediately counted and analyzed. Consideration should be given to chemical compatibility between the sample and the counting container. A leaking sample could seriously damage an expensive detector system.

Sample size:	300 ml
Preparation:	None
Counting Technique:	HPGe
Counting Time:	120 minutes
Estimated MDA:	MDA's will vary greatly from sample to sample and nuclide to nuclide, depending on the sample matrix and interfering nuclides. The following list of MDA's is representative. These were determined from a water blank at LLNL (see Table I).

### Solids

Solid samples are also easy to measure by spectroscopy, although drying, grinding, pressing or cutting may be necessary to reduce the sample to a known geometry.

Sample size:	300 g
Preparation:	None

TABLE I  
List of MDA's

Nuclide	MDA (pCi/mL)	Nuclide	MDA (pCi/mL)
$^7\text{Be}$	0.2	$^{147}\text{Pm}$	600
$^{57}\text{Co}$	0.08	$^{152}\text{Eu}$	0.08
$^{60}\text{Co}$	0.02	$^{226}\text{Ra}$	0.06
$^{95}\text{Zr}$	0.03	$^{235}\text{U}$	0.04
$^{137}\text{Cs}$	0.03	$^{241}\text{Am}$	0.1

Counting Technique:	HPGe
Counting Time:	120 minutes
Estimated MDA:	MDA's will vary from sample to sample and nuclide to nuclide, depending on the sample matrix and interfering nuclides. The following list of MDA's is representative. These were determined from a water blank at LLNL, calculated with self-attenuation as that of soil (see Table II).

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TABLE II  
List of MDA's

Nuclide	MDA (pCi/g)	Nuclide	MDA (pCi/g)
$^7\text{Be}$	0.1	$^{147}\text{Pm}$	400
$^{57}\text{Co}$	0.04	$^{152}\text{Eu}$	0.05
$^{60}\text{Co}$	0.01	$^{226}\text{Ra}$	0.03
$^{95}\text{Zr}$	0.02	$^{235}\text{U}$	0.02
$^{137}\text{Cs}$	0.02	$^{241}\text{Am}$	0.08