ENTERED

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

SEP 4 2012

OFFICE OF AIR AND RADIATION

Mr. J. R. Stroble
Manager, National TRU Program
Carlsbad Field Office
U.S. Department of Energy
P.O. Box 3090
Carlsbad, NM 88221-3090

Dear Mr. Stroble:

This letter transmits the results of the U.S. Environmental Protection Agency's (EPA's) evaluation of the Analytical Chemistry Laboratory (ACL) located at the U.S. Department of Energy's (DOE's) Argonne National Laboratory (ANL). On July 31-August 1, EPA performed this evaluation to determine whether the laboratory conducts radiological characterization of transuranic (TRU) waste in conformance with standard laboratory practices employed by analytical laboratories around the DOE Complex and analytical requirements dictated by DOE's WIPP Program. Our evaluation found numerous technical deficiencies which are discussed in the enclosed report (A-98-49; II-A4-165).

As discussed at the exit meeting, no data generated by the ACL after July 31, 2012, can be used by ANL-CCP to characterize WIPP-destined TRU waste. Additionally, the EPA-identified deficiencies are severe enough that CBFO and the Central Characterization Project (CCP) responsible for characterizing ANL TRU waste may not use ACL's services for analyzing TRU waste until:

- ACL conducts a thorough assessment of its radiochemical laboratory practices and addresses deficiencies and the EPA-identified technical issues; and
- EPA approves ACL's TRU waste-specific laboratory operation prior to ACL analyzing any TRU waste destined for WIPP disposal.

Since 2007, the ACL has been assaying remote-handled (RH) transuranic (TRU) waste for the Central Characterization Project (CCP), responsible for implementing DOE's TRU waste characterization program at ANL. At the time of EPA's evaluation, ACL was not analyzing any RH TRU waste. Therefore, EPA focused on interviewing laboratory management and personnel, as well as reviewing laboratory records that documented the operational aspects of spectrometric and radiometric equipment. EPA staff also reviewed other pertinent laboratory activity records, such as laboratory notebooks, logbooks, technical memoranda, equipment maintenance and performance records and electronic data files.

If you have any questions regarding this evaluation, please contact Rajani Joglekar at (202) 343-9462 or Ed Feltcorn at (202) 343-9422.

Sincerely,

Tom Peake

Tom Peake, Director
Center for Waste Management and Regulations

Enclosure

cc: Electronic Distribution
Christine Gelles, DOE EM
Doug Tonkay, DOE EM
Alton Harris, DOE EM
Joe Franco, CBFO
Marcus Pinzel, CBFO NTP
Courtland Fesmire, CBFO QA
Dale Dietzl, DOE CH
D K Ploetz, WTS-CCP
Irene Quintana, CCP
Trais Kliphuis, NMED
Tim Hall, NMED
Site Documents
Jon Edwards, EPA
Alan Perrin, EPA
Ray Lee, EPA

DOCKET NO: A-98-49, II-A4-165

WASTE CHARACTERIZATION REPORT

EPA EVALUATION OF THE ANALYTICAL CHEMISTRY LABORATORY AT THE ARGONNE NATIONAL LABORATORY

August – September 2012

U.S. Environmental Protection Agency
Office of Radiation and Indoor Air
Center for Waste Management and Regulations
1200 Pennsylvania Avenue, NW
Washington, DC 20460

September 2012

TABLE OF CONTENTS

ATTACHMENTS

ACRONYMS AND ABBREVIATIONS

ACL	Analytical Chemistry Laboratory
Al	aluminum
Am	americium
amu	atomic mass unit
ANL	Argonne National Laboratory
ANL-E	Argonne National Laboratory-East
ANL-W	Argonne National Laboratory-West
ASTM	American Society for Testing and Materials
BDR	batch data report
Bi	bismuth
CBFO	Carlsbad Field Office
CCP	Central Characterization Project
Cd	cadmium
CFR	Code of Federal Regulations
Cm	curium
cpm/dpm	counts per minute per disintegrations per minute
Cs	cesium
DIW	deionized water
DOE	U.S. Department of Energy
E_{max}	maximum energy of beta emission
EPA	U.S. Environmental Protection Agency
Eu	europium
FWHM	full width at half maximum
g	gram
GPC	gas proportional counting
HP	health physics
ICP-MS	inductively coupled plasma–mass spectrometry
INL	Idaho National Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
keV	kiloelectronvolts
LBNL	Lawrence Berkeley National Laboratory

LCS	laboratory control sample
LOQ	limits of quantitation
LOQI	list of qualified individuals
megohm/cm	megohms per centimeter
MeV	megaelectronvolts
mg	milligram
mg/cm^3	milligrams per cubic centimeter
mL	milliliter
NCR	nonconformance report
NIST	National Institute of Standards and Technology
ORIA	Office of Radiation and Indoor Air (EPA)
Pu	plutonium
QA	quality assurance
QAO	quality assurance objective
QC	quality control
RH	remote-handled
ROI	region of interest
RPD	relative percent difference
Sn	tin
SOP	standard operating procedure
SPM	site project manager
Sr	strontium
T1	Tier 1
TRAMPAC	Trupact Authorized Methods for Payload Control
TRU	transuranic
U	uranium
U_{Nat}	natural uranium
WIPP	Waste Isolation Pilot Plant
WTS	Washington TRU Solution
Y	yttrium

1.0 INTRODUCTION

This report presents the results of the U.S. Environmental Protection Agency's (EPA's) evaluation of the Analytical Chemistry Laboratory (ACL) located at the U.S. Department of Energy's (DOE's) Argonne National Laboratory (ANL).[1] EPA performed this evaluation to determine whether ACL's radiological characterization of transuranic (TRU) waste conforms with the analytical requirements of DOE's Waste Isolation Pilot Plant (WIPP) program and is consistent with standard laboratory practices employed throughout the DOE complex. ACL[2] assayed samples of remote-handled (RH) debris to support the development of radionuclide-specific scaling factors for RH TRU wastes from the K-Wing at ANL.

At the time of this EPA evaluation (July 31–August 1, 2012), the ACL was not analyzing RH TRU samples and no future WIPP samples were scheduled. Therefore, EPA focused on interviewing laboratory management and personnel and reviewing laboratory records that documented the operational aspects of spectrometric and radiometric equipment and other pertinent laboratory activities, such as laboratory notebooks, logbooks, technical memoranda, equipment maintenance and performance records and electronic data files. In 2010, EPA had conducted a similar evaluation of the Idaho National Laboratory's (INL) analytical laboratory[3] that analyzed RH WIPP samples to support radionuclide scaling factors. EPA concluded that INL's analytical laboratory program was in conformance with WIPP requirements (Docket No. A-98-49; II-A4-130, August 2010).

During this evaluation, EPA identified two concerns with identical technical issues (see Attachment C at the end of this report) relative to ACL's operations. The first concern (Issue No. ANL-CCP-RH-ACL-2012-01CR) is directed to CBFO and states that CBFO and/or CCP may not use ACL's services for analyzing TRU waste destined for WIPP disposal until (1) ACL identifies and addresses operational deficiencies (including those mentioned in the report), and (2) EPA approves ACL's TRU waste-specific laboratory operation prior to ACL analyzing any future WIPP samples. In addition, as a result of this concern, no data generated by ACL after July 31, 2012, can be used to characterize WIPP-destined TRU waste. The second concern (Issue No. ANL-CCP-RH-ACL-2012-02C) presents only the technical issues specific to ACL that encompass several aspects of the analytical processes at ACL (categorized in five areas), based on EPA's sample. For ACL to begin analyzing WIPP-destined TRU waste, ACL must conduct a thorough assessment of its radiochemical laboratory to identify deficiencies and address those deficiencies as well as the EPA-identified technical issues. Once ACL has completed this step,

---

[1] ANL was initially designated "ANL-E" (ANL-East) to distinguish it from ANL-West (ANL-W). Since 2009, ANL-W has been renamed the Materials and Fuel Complex (located in Idaho Falls, Idaho), and ANL-E is simply ANL (located in Illinois).

[2] For more than 5 years, ACL has been responsible for sampling and analyzing TRU waste destined for disposal at WIPP for the Central Characterization Project (CCP). Before tasking ACL with RH TRU waste analysis, CCP evaluated and was satisfied with the laboratory's activities that supported radiological characterization of RH TRU waste. Similarly, the Carlsbad Field Office (CBFO) has certified ACL for sampling and analyzing RH TRU waste generated at ANL.

[3] In 2010, the INL analytical laboratory was the Idaho Nuclear Technology and Engineering Center (INTEC) Laboratory; it has since been renamed.

EPA inspection and approval of the ACL laboratory operation are necessary before ACL can analyze the WIPP-destined RH TRU waste and before CBFO can use the resultant data for radiological characterization.

Since EPA's initial RH approval in 2007, EPA approved two Tier 1 (T1) changes to the ANL ANL-CCP RH TRU characterization program for K-Wing wastes: (1) Docket No. A-98-49; II-A4-132, in September 2010 for K-Wing debris; and (2) Docket No. A-98-49; II-A4-162, in June 2012 for K-Wing solidified liquids. The scaling factors for both changes were developed using radiometric and spectrometric data from ACL, which had not been formally approved by EPA.

This report serves as EPA's public notification of the results of EPA's evaluation of ACL. This information will be provided through the EPA website and by emails to the WIPPNEWS list, in accordance with Title 40 of the Code of Federal Regulations (40 CFR) 194.8(b)(3).

2.0 PURPOSE OF THIS REPORT

This report presents the results of EPA's evaluation of the technical activities performed by ACL in support of ANL-CCP's characterization of RH TRU wastes. This report presents the technical basis and results of EPA's evaluation of analytical data from ACL, which have been conveyed to DOE separately by letter. The DOE documents that EPA reviewed for this evaluation are listed in Attachment B. Copies of these documents can be requested from the following address:

Manager, National TRU Program
Carlsbad Field Office
U. S. Department of Energy
P O Box 3090
Carlsbad, NM 88221-3090

3.0 SCOPE OF THIS EVALUATION

The scope of this evaluation is the laboratory operations that supported the development of radionuclide-specific scaling factors for WIPP-bound RH TRU wastes. Sections 6.1 and 6.2 of this report detail the two technical areas assessed during this evaluation:

- General laboratory operations and inductively coupled plasma–mass spectrometry (ICP-MS).
- Radiometric analyses.

This evaluation focused exclusively on activities conducted by the laboratory. In lieu of inspection checklists, EPA prepared specific questions, which were provided to ACL in advance of the evaluation and are included in this report.

4.0 EVALUATION PERSONNEL

Table 1 lists the EPA evaluation team members and the personnel contacted, along with their affiliations and technical areas of expertise. This list includes personnel present at meetings conducted as part of this evaluation.

Table 1. Tier 1 Evaluation Personnel

Personnel Name	Affiliation	Area of Expertise, Function
Rajani Joglekar	U.S. EPA ORIA	Lead Inspector
Ed Feltcorn	U.S. EPA ORIA	Inspector
Patrick Kelly	U.S. EPA, SC&A	Technical Inspector
Dorothy Gill	U.S. EPA, SC&A	Technical Inspector
Dale Dietzel	Argonne Site Office, DOE	Deputy Federal Program Director
Susan Heston	DOE Argonne Site Office	Observer
Vivian Sullivan	ANL ACL	Manager ACL, Analyst
Dan Pancake	ANL	Project Manager, Nuclear Operations Deactivation, Demolition & Decommissioning Program
	WTS-CCP	Observer
Irene Quintana	WTS-CCP	RH Manager
Donald Graczyk	Argonne ACL	ACL ICP-MS Chemist
Andrew Gabel	DOE-Argonne Site Office	Alpha Gamma Hot Cell Facility, Federal Program Director
Yifen Tsai	ANL	ICP-MS Analyst
Susan Lopykinski	ANL	QA/QC Coordinator
Marcus Pinzel	CBFO DOE	RH Manager

6.0 TECHNICAL EVALUATION

Evaluation Approach

ACL was not operational during EPA's evaluation. The laboratory had recently been relocated to a room that was still in preparation at the time of the evaluation. The radiometric equipment and ICP-MS were operational, although they were not actively assaying WIPP samples. EPA was aware of this prior to the evaluation and focused on documentation of laboratory practices and interviews with laboratory personnel. ACL provided the most recent WIPP batch data report (BDR), SDG:TIC-1, and a previously submitted WIPP BDR (7TRUA50-A) to serve as guides for the evaluation. Although EPA did not directly assess the acceptability of these BDRs, they were used as objective evidence for the laboratory's operations and practices that were relevant to TRU wastes.

EPA chose a sample of ACL activities for this evaluation and evaluated those activities based on the standard operating procedures (SOPs) provided, general laboratory practices, documentation and information provided by the laboratory personnel during the evaluation. Specifically, EPA prepared a series of questions based on comparing the two WIPP BDRs, a nonconformance report (NCR), two SOPs used for WIPP samples and the Supplemental Quality Assurance Plan. EPA provided these questions to ACL in advance of the evaluation. EPA's questions and the responses provided by ACL personnel are shown in Attachment A, Table A-1. Table A-1

presents EPA's analytical inquiry into ACL's (1) general laboratory practices and ICP-MS and (2) radiometric analyses.

Laboratory Documents Provided

EPA evaluated ACL SOPs, BDRs, reports, logbooks and memoranda that documented the sample preparation, spectrometric and radiometric operations of the laboratory. Attachment B lists all of the documentation reviewed before or during the onsite evaluation.

6.1 General Laboratory Practices and ICP-MS

Attachment A, Table A-1, summarizes EPA's questions and ACL responses with respect to ACL general laboratory practices and ICP-MS. Tables A-1 and A-2 in Attachment A contain duplicative information, as indicated.

ACL processes a variety of sample media in support of several federal programs. The WIPP samples analyzed thus far have been smears and fluids; the amount of actual chemical processing of these media is minimal. When samples have high associated external exposure (i.e., dose) rates, the hot cell personnel perform what amounts to sample preparation, in that they collect small volumes of fluid that require only dilution prior to analysis. When this is the case, the sample preparation must be done "according to client work plan and procedures," according to SOP:ACL-274. The only additional preparation for such samples is adjustment of pH and subsequent dilution. This is significant because in such cases it is important to accurately dispense small, specific volumes with a high degree of reproducibility.

EPA identified instances of technically inadequate practices, incomplete or missing documentation, technically adequate SOPs not being followed, and incomplete or undocumented training, as indicated in Table A-1. These instances were in all of the areas evaluated during this evaluation. Additionally, the ACL analysts that EPA interviewed were not aware of several important requirements of the WIPP analyses. Specifically, when interviewed during this evaluation, the ACL personnel (Yifen Tsai and Susan Lopykinski) who conducted the ICP-MS analyses were not aware of many WIPP requirements, as detailed in Table A-1.

Of particular importance to EPA were the lack of measurement control and independent verification. There were several instances in which technically based (i.e., statistical) acceptance criteria were not developed for fundamentally important aspects of ICP-MS measurements, such as performance of quality control (QC) samples and standards, and uncertainty for internal standards and isotopic dilution. In both of these areas, the acceptability of ICP-MS data is left to the judgment of the individual analyst. Additionally, records for the preparation of internal standards were not maintained. It is unclear exactly how analysts can have confidence in ICP-MS measurements without such criteria.

Equally important is the lack of an independent means to verify the accuracy of the actinide measurements, since a single [uranium (U)] standard is used for all actinides. The lack of independent measurements does little to support the laboratory's accuracy for actinide determinations using ICP-MS. This affects others areas also, because the ICP-MS data are used

in combination with radiometric-derived gross alpha and alpha spectrometry values to generate concentrations for specific WIPP-tracked radionuclides [i.e., americium-241 (^{241}Am); see section 6.2].

EPA concluded that ACL operations were not adequate in the majority of the areas related to laboratory operations and ICP-MS that were evaluated. Details regarding EPA's concern are presented in section 6.3.

6.2 Radiometric Equipment and Documentation

Analytical Approach

ACL used the following analytical approach for radionuclides in WIPP samples:

- Chemical separation and radionuclide-specific measurements are not performed for plutonium-238 (^{238}Pu), ^{239}Pu, ^{240}Pu and ^{241}Am; instead, a combination of gross alpha values form gas proportional counting (GPC), an alpha scan from alpha spectrometry (in which spectra do not differentiate radionuclide-specific peaks, e.g., ^{238}Pu and ^{241}Am) and ^{241}Am values from gamma spectrometry and isotopic assigments based on ICP-MS are manipulated to produce quantitative values for specific radionuclides.
- ^{241}Pu is quantified by subtracting the gamma-derived ^{241}Am value from the ICP-MS-derived ^{241}Am/^{241}Pu value.
- Strontium-90 (^{90}Sr) is quantified by (1) subtracting the values for known beta emitters determined by another method from the gross beta value obtained by GPC, i.e., ^{241}Pu (described above), (2) subtracting the gamma-spectrometry-derived values for cesium-134 (^{134}Cs), europium-154 (^{154}Eu) and ^{137}Cs values from the GPC gross beta, and (3) assigning the remainder to ^{90}Sr.

This is a non-standard approach; i.e., it is not consistent with industry consensus standards. Specifically, EPA was concerned about the approach for the alpha scan and ^{90}Sr procedures:

- The alpha scan does not involve an elemental separation and internal tracers or carriers. As a result, the spectral data contain overlapping peaks for WIPP-tracked radionuclides (e.g., ^{238}Pu and ^{241}Am). It is not clear how quantitative this technique can be since the lack of internal tracers prevents correction for losses.

- The value for ^{90}Sr is obtained by subtracting known beta/gamma-emitters that were identified by gamma analysis from the gross beta value or from additional information of the sample's origin. The remaining beta activity is assigned to ^{90}Sr. It is not clear that this method provides radionuclide values that truly represent the ^{90}Sr content of the samples.

Because ACL's approach was nonstandard, EPA requested documentation that this approach was adequate, i.e., independent analyses to verify that the approach provided technically adequate radionuclide-specific values. Vivian Sullivan stated that the ACL method was developed by the laboratory and that it is a "process developed over time" that

is "only performed by analysts with experience in the technique." ACL did not provide documentation of the analysts' training of the technique, nor had it performed or documented an independent verification of this approach. Given the approach's lack of specificity with regard to the alpha scan and ^{90}Sr analyses and the lack of independent verification, there is no objective evidence that this approach produces valid data for specific WIPP-tracked radionuclides. ACL personnel agreed that they would be able to perform and document some type of validation of this method in the future.

Measurement control and the lack of an independent means to verify the accuracy of radionuclide measurements were a concern for radiometric analyses. Many of the radionuclide standards were old; preparation of standard counting geometries was inadequately documented; and several standard counting geometries had been prepared 20 years ago and were still in use without any independent verification that they continue to be adequate.

The logbook pages from October 1999 that documented the activity for the alpha standards composed of ^{238}U, ^{234}U, ^{239}Pu and curium-243 (^{243}Cm) were difficult to understand. Starting with the pages that documented the solutions' initial preparation and dilution by ACL, EPA was unable to duplicate the values for the specific radionuclides listed. ACL personnel were unable to provide a definitive explanation of the standards' activity and stated that the standards' use predated the current ACL employees' tenure at ACL. Despite these issues, ACL used these alpha standards to determine the adequate response of instruments, without independent corroboration.

Counting Equipment

The ACL radiometric equipment used for WIPP analyses is listed below:

- Multi-Detector Tennelec Gas Proportional Counter, LB-4000, with eight detectors, used for gross alpha-beta.
- High purity germanium gamma detectors, four stand-alone systems with separate electronics and computers, used for gamma emitting radionuclides.
- Alpha spectrometers, 14 used for WIPP analyses stored in one cabinet with common electronics and computer, used for the alpha scan analyses.

ACL had additional gamma systems, alpha spectrometers and a liquid scintillation counter, but EPA did not evaluate these because they were not used for WIPP analyses. During the evaluation, EPA asked a series of oral questions specific to radiometric analyses that focused on the calibration, maintenance and operation of counting equipment, summarized in Appendix A, Table A-2. (Tables A-1 and A-2 contain some duplicative information, as indicated.) EPA requested and obtained specifications for all systems, as well as other documentation relevant to the systems' calibration, maintenance and use. EPA found a general lack of rigor in instrument calibration and maintenance; documentation of these activities was inconsistent in some cases and absent in others. EPA observed that the age of the radionuclide standards and the lack of verification using independent sources were concerns for the radiometric equipment. EPA's concerns in this regard are summarized in section 6.3.

6.3 EPA Concerns

EPA combined the concerns from laboratory operations, ICP-MS and radiometric analyses into two overall concerns regarding ACL's adequacy for performing WIPP analyses. The main aspects of the concerns fall into five areas, as shown below:

Measurement Control

- Lack of technically based (statistical) control limits.
- Lack of independent checks.
- Lack of control of support equipment (pipets, laboratory water).
- Inadequate control of instrument performance (plateau checks when changing P-10 gas in GPC).
- Inadequate records of calibration and performance-related aspects.

Records

- Lack of records for several important laboratory areas and functions.

Training

- No training in the revised SOPs.
- No training in the quality assurance (QA) manual.
- No records of training in the supplemental QA requirements for this project.

Written Procedures (SOPs)

- Technically inadequate.
- Incomplete.
- Do not document actual practices.
- Were routinely not followed.

Communication of WIPP-Specific Requirements to Analysts

These concerns are summarized on the EPA Inspection Issue Tracking Forms in Attachment B and were discussed with ACL and CBFO personnel at the evaluation closeout meeting at ACL on August 1, 2012.

7.0 FINDINGS OR CONCERNS

During this evaluation, EPA identified two concerns that encompassed several aspects of the analytical process at ACL, as discussed in this report. The concerns encompassed all aspects of ACL's operation that were within EPA's evaluation scope.

8.0 CONCLUSIONS

As a result of this evaluation, CBFO and/or CCP may not use ACL's services for analyzing TRU waste destined for WIPP disposal until (1) ACL identifies and addresses operational deficiencies (including those mentioned in the report), and (2) EPA approves ACL's TRU waste-specific

laboratory operation prior to ACL analyzing any WIPP samples. In addition, no data generated by ACL after July 31, 2012, can be used to characterize WIPP-destined TRU waste.

For ACL to begin analyzing WIPP-destined TRU waste, ACL must conduct a thorough assessment of its radiochemical laboratory to identify deficiencies and address those deficiencies as well as the EPA-identified technical issues. Once ACL has completed this step, EPA inspection and approval of the ACL laboratory operation are necessary before ACL can analyze the WIPP-destined RH TRU waste and before CBFO can use the resultant data for radiological characterization.

ATTACHMENT A
EPA EVALUATION QUESTIONS AND ACL RESPONSES

Table A-1. EPA Evaluation Questions and ACL Responses, July 31–August 1, 2012

Question No.	Document/Section	EPA Question or Information Request/ Answer Derived from EPA Evaluation July 31–August 1, 2012[a]
BDR 7TRUA50-A		
1	Section 2, page 4, 2nd paragraph	**EPA Question (EPA):** This passage states, "A pipet calibration checked by the ACL was used to sample the aliquot provided to the ACL." Please clarify if the intent was to say that a calibrated pipet was used. **Answer Derived from EPA Evaluation (Answer):** ACL has a pipet calibration SOP, ACL-145, but does not use it as written and does not have records of data used to assess the pipets.
2	Page 5, 1st and 2nd paragraphs	**EPA:** Please provide the basis for the calculations of ^{238}Pu and ^{90}Sr. **Answer:** ACL provided the derivation of the calculations for these radionuclides.
3	Section 3, Quality Control Results	**EPA:** Please specify which analytical techniques generated the relative deviations stated. **Answer**: The ICP-MS technique does not use an LCS, so the BDR should have specified what analytical techniques this statement applies to.
4	General	**EPA**: Please provide a summary of LCS results to support the statement "Lab control samples were satisfactory," including acceptance criteria used. Note: if clear, unambiguous summaries, with acceptance criteria, are available in the BDR for each analytical technique, please provide page numbers. **Answer**: The BDR does not provide acceptance criteria.
5	General	**EPA**: Provide summaries, or BDR references to summaries, for duplicate/replicate results for all techniques (as above). **Answer**: According to NCR-ANL-2344-11 from BDR 7TRUA50-A, duplicates were not analyzed even though required by the project plan (in the supplemental requirements).
NCR-RHANL-2344-11		
1	General	**EPA**: The SPM originally signed off the batch on 11/22/2011, and the NCR was not initiated until 12/21/11. Who identified the NCR and under what circumstances? **Answer**: Irene Quintana said that she identified the NCR when reviewing the results. By then, the

		BDR had already been through the SPM-level review, which missed the duplicate issue. The BDR was reviewed by Irene Quintana and Jene Vance; they noticed the problem with duplicates, so ACL initiated an NCR. The NCR root cause analysis performed does not address the real cause. It was too late to reanalyze the samples because reporting of the data was delayed due to some other work. Data had been through ACL review, but this review did not identify that the data did not meet the requirements of the Sampling and Analysis Plan.
2	Section 7b	**Background:** Section 7b of the NCR states, "CCP-TP-512, Revision 5, CCP Remote-Handled Waste Sampling, Section 2.4, Quality Assurance Objectives (QAO's), 2.4.1 Precision: Sampling precision is established by comparing the relative percent difference (RPD) between duplicate samples. A Nonconformance Report (NCR) shall be issued for any duplicate samples with RPD's greater than 25 percent." DOE-WIPP-02-3214, Revision 2, Table 4.3, states, "Laboratory Duplicate - A laboratory duplicate is analyzed at least once per analytical batch. A laboratory duplicate is a separate aliquot from the same field sample carried through the entire analytical procedure." Section 7c, Actual Condition, states, "There was no Laboratory duplicate created because of the presence of several field duplicates, however, the field duplicates do not all meet the required RPD limit of less than 25%." Section 19, Final Disposition, technical justification, states, "Laboratory duplicate was not performed due to the numerous field duplicate samples submitted for analysis. The RPD for field duplicates samples did not meet the criteria due to inhomogeneous nature of the samples." ANL CA611-02-01 states, "For ACL job 10-0059 (K-Wing analysis), replicate samples were drawn from each liquid container. Per SOP ACL-274 R.2, this could be counted as the duplicate sample for analysis, and was considered as such by the ACL. Upon discussion with the client after completion of the analytical work and reporting, the degree of potential inhomogeneity of the sample made these duplicates more useful as field duplicates and triplicates for their calculations. Therefore, these samples did not count as our laboratory duplicates and we did not perform a sample split on a single aliquot before dilution to create a different laboratory duplicate." **Questions:** **EPA**: Please clarify the underlined text above.

		Answer: CCP's justification in section 19 of the NCR cannot be supported because only one sample had more than one layer. **EPA**: Please provide the exact reference in SOP ACL-274 where it states that a field duplicate may be used as a laboratory duplicate. As these duplicates have different functions, it is unclear how or why they were apparently used interchangeably. **Answer**: The SOP is incorrect and needs to be revised.
ACL-271, Determination of Trace Elements/Isotopes in Solution by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Using the PerkinElmer SCIEX ELAN DRC II		
1	Sections 5.2 and 5.3	**EPA**: Section 5.2 refers to "Volumetric flasks of suitable precision and accuracy," and section 5.3 refers to "Volumetric pipets of suitable precision and accuracy." Please define what accuracy and precision requirements are attached to the pipets and volumetric flasks, how these requirements are met and where these activities are documented. **Answer**: For pipets, ACL does check accuracy but does not check precision. ACL has an SOP for pipet calibration/verification but does not follow it. ACL does not keep records of measurements and so cannot support the volumes used, which, in turn, means that it cannot support quantitation.
2	Section 6.3	**EPA**: Section 6.3 of ACL-271 requires "Laboratory de-ionized water having a specific resistivity of at least 17.5 megohm/cm," but section 6.1 of ACL-274 requires "Deionized water (DIW), Type II." Please explain how the laboratory accommodates two different requirements for water quality used in testing. **Answer**: The deionized water system has an inline conductivity/resistivity meter that is not independently checked. This means that ACL cannot support use of water of the required quality.
3	Section 7.1	**EPA**: Section 7.1 states, "If a sample is assigned an alias...." Please explain. **Answer**: ACL provided an adequate explanation.
4	Section 7.2.1	**EPA**: Section 7.2.1 describes the use of internal standards but does not identify what compound(s) are used. Please provide references where this information is located. **Answer**: The laboratory could not provide internal standard preparation records. ACL uses a recipe but does not keep individual preparation records (e.g., lot numbers for nitric acid), so there is incomplete traceability for the sample preparation records.
5	Section 8.1	**EPA**: Section 8.1 describes what instrument checks are performed but does not provide pass/fail acceptance criteria for these QC analyses. Please provide references where this and the QC

		acceptance criteria for this method are documented. **Answer**: Performance checks have no objective acceptance criteria, and their acceptability is based solely on the analyst's experience. If this system is used, the text regarding this practice should be taken out of the SOP. However, the laboratory should statistically calculate or assign objective acceptance criteria to ensure consistency of data interpretation.
6	Section 8.2.1	**EPA**: Section 8.2.1 states, "Instrumental drift may be corrected by normalization to an internal standard present in the blanks, standards, and samples." Please describe the situation or circumstances when this is used and when it is not. **Answer:** ACL does use internal standard normalization for WIPP samples, but the SOP needs to specifically state what is done to achieve this.
7	Section 8.2.1, p. 6	**EPA**: Section 8.2.1, page 6, states, "The uncertainty of results obtained by quantitative analysis is estimated to be ±10%." Please identify the basis and mechanism for this determination and where it is documented. **Answer**: ACL has plenty of data but does not calculate real uncertainty limits. The laboratory does not construct control charts, so it does not have any indication of trends within the data.
8	Section 8.2.1, p. 6	**EPA**: Section 8.2.1, page 6, states, "This level of accuracy is qualified with analysis of QC solutions." Please explain the meaning of "qualified" in this sentence and provided the calculations supporting the "qualification." **Answer**: ACL did not respond to this question.
9	Section 8.2.1, p. 6	**EPA**: Section 8.2.1, page 6, states, "If the analysis of a QC solution results in a relative error ≥±10% the analyst must recalculate the estimated uncertainty for the samples based on maximum relative error of the calculated results for the bracketing QC solutions and record this on the data summary report." a. Please explain how the estimate is recalculated. b. Please identify where the results of this activity are documented. c. Please explain why corrective action is not initiated in response to "failed" QC. **Answer**: In practice, this procedure is only used if the analyst runs out of sample, but the SOP does not indicate that. Instead, the text in the SOP appears to indicate that this can be done any time a QC sample fails. ACL needs to revise the SOP.
10	General	**EPA**: Please provide the formula used to calculate relative error. **Answer**: ACL provided an adequate formula.

11	Section 8.2.2, Surrogate Quantitative Analysis	**EPA**: Surrogate Quantitative Analysis: If this process was used for any samples in BDR 7TRUA50-A: a. Please identify which samples and what surrogates were used. b. Please provide validation data for surrogate quantitative analysis on WIPP samples. **Answer**: Basically, ACL uses the U standard as a surrogate for quantitation of other actinides. Don Graczyk provided written and verbal information and a copy of ASTM C1590-04 (2009), which provides evidence that this technique works. However, the laboratory does not perform its analysis exactly according to the ASTM standard; therefore, it needs to have some sort of validation that the technique works as implemented. ACL needs to get a second source U standard and second source thorium standard.
12	Section 8.2.2.3	**EPA**: Section 8.2.2.3 states, "The accuracy of the surrogate-quantitative analysis determination can be inferred to be the relative error calculated from a quantitative-analysis determination of a NIST traceable element with a mass within 10 amu of the nuclide of interest." Please explain why accuracy is "inferred" rather than determined or calculated. **Answer**: ACL did not provide any information in response to this question.
13	Section 8.2.3	**EPA**: Please explain how the uncertainties of ±5% and ±1% for standard addition and isotope dilution were determined. **Answer**: These uncertainties are assigned values, not statistically calculated.
14	Section 8.3.3	**EPA**: Section 8.3.3 states, "Report results above the LOQ (limits of quantitation) to no more than three significant figures." Please clarify if results are always reported to three significant figures or, if not, how and by whom this reporting decision is made and where this is documented. **Answer**: The laboratory was unable to answer this question.
ACL-274, Isotopic Analysis of Samples for Disposal at WIPP		
1	Section 9.6	**EPA**: Section 9.6 of AL-274 states that "Acceptable limits of blanks, laboratory control samples/calibration check samples, duplicates and matrix spikes and duplicates will be detailed in the client work plan." No acceptance limits could be found in the Supplemental Quality Assurance Project Plan, 10-0059. **Answer**: ACL does not appear to have objective acceptance criteria for QC samples. Blanks are judged subjectively by analysts. Standards have assigned acceptance criteria but they are not statistically based. The development of acceptance criteria needs to be formalized and its statistical basis calculated and documented.

		EPA: Quality Assurance Plan, C0030-0221, section 5.3, states, "Control limits on QC samples such as...are defined in SOP's along with actions taken if controls indicate potential problems." The limits could not be located as stated above. **Answer**: The SOPs do not contain any limits. **EPA**: Please identify where QC limits are specified for all the analytical techniques used for data reported in BDR 7TRUA50-A. **Answer**: The laboratory did not provide this information to EPA.
2	General	**EPA**: Please clarify if SOP ACL-258 or any part thereof was used to generate data for BDR 7TRUA50-A. Also please confirm what other ACL SOPs (if any) were used to generate the data for BDR 7TRUA50-5. **Answer**: Only SOP ACL-274 was used.
3	Section 2.1	**EPA**: Section 2.1 states, "The sample is prepared according to the client work plan." The work plan does not include sample preparation instructions. Please verify that the sample preparation instructions contained in section 8 of ACL-274 are those used for WIPP samples. **Answer**: ACL verified that sample preparation instructions in ACL-274, section 8, are used for WIPP samples. This is adequate.
4	Sections 8.2.1 and 8.2.3	**EPA**: Section 8.2.1 states, "Samples may be prepared for gamma analysis by the client as the initial aliquot." Section 8.2.3 states, "Samples may be similarly prepared by an ACL analyst." Please clarify what this means in terms of who actually prepares samples and where this is documented. **Answer**: The laboratory provided calibrated pipets and observed operations. Pipets are not calibrated in accordance with ACL SOP 145, and there are no records of any calibration. The hot cell sample collection personnel prepared the samples for "as low as is reasonably achievable" purposes; i.e., the samples had elevated external dose rates.
5	Section 8.3.7	**EPA**: What amount of activity is "too low to allow direct plating"? **Answer**: Vivian Sullivan says ~1 nanocurie is the goal.
6	Section 8.6	**EPA**: Please identify the source(s) for the physical constants (radionuclide half-lives, photon transition probabilities, specific activity, etc.) when performing the calculations in this section. **Answer**: Lawrence Berkeley National Laboratory (LBNL) data are used, not TRAMPAC values.
7	Section 8.6.1	**EPA**: Please explain the technical basis, and provide the validation information for, the data

		manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
8	Section 8.6.2	**EPA**: Please explain the technical basis, and provide the validation information for, the data manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
9	Section 8.6.3	**EPA**: Please explain the technical basis, and provide the validation information for, the data manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
10	Section 8.6.4	**EPA**: Please explain the technical basis, and provide the validation information for, the data manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
11	Section 8.6.5	**EPA**: Please explain the technical basis, and provide the validation information for, the data manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
12	Section 8.6.6	**EPA**: Please explain the technical basis, and provide the validation information for, the data manipulations contained in this section. **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
13	Section 8.6.8	**EPA**: Please identify the criteria used to evaluate if the "actinides in the sample are high compared to the fission products." **Answer**: The technical bases for the data manipulations are not addressed in detail in any ACL document. ACL did not provide any validation information for this approach.
14	Appendix A, section 1.1.3	**EPA**: The text states, "If contamination is evident, the room and/or detector should be examined and cleaned, if possible; the background should be repeated until it is at an acceptable level." a. Please explain what actions are taken if cleaning the room and/or detector is not possible.

		Answer: Vivian Sullivan stated, "If the background issue could not be resolved with cleaning and HP [health physics] coverage, the instrument would be taken out of services until the spot of contamination was detected and remediated." Also, "Repeating the background ensures that a background with one high value is not used, which would lead to underreporting of that isotope." The oral response is not consistent with what is stated in the SOP. The SOP's language does not adequately explain the appropriate actions that should be taken. The SOP should state the equivalent of "background should be repeated until it is at an acceptable level."
15	Appendix A, section 1.1.3	**EPA**: The text states, "The acceptance of the background is based on historical patterns; some detectors have less shielding and so intrinsically higher backgrounds." Please provide the objective criteria used when assessing the acceptability of detector backgrounds and the location of the documentation for these actions. **Answer**: It appears that the background is judged on subjective criteria. This does not provide a basis for consistent interpretation of the same data set.
16	Appendix A, section 1.2.2	**EPA**: The text states, "The percent deviation from the standard reference value will be calculated; more than ±5% deviation is a warning and ±10% requires examination of the calibration." a. Please explain the genesis of the acceptance criteria. b. Please explain what is done during examination of the calibration. c. Please identify the location of the guidance regarding the corrective actions and their documentation. **Answer**: Vivian Sullivan stated, "The genesis of the 5%/10% predates my work in the ACL, so I don't know the specifics of the choice. These criteria were acceptable to WIPP/CCP when we were originally audited, as the values have not changed. The spectrum is run and analyzed with GammaVision and the T-0 corrected [decay-corrected] values are compared with the reference values for the standard, ACL SOP-166." This practice is technically unsupportable; acceptance criteria must be statistically determined to ensure that default values are good.
17	Appendix A, section 1.2.3	**EPA**: The text states, "QA check counts shall be run" but does not specify how these checks are used to assess the correctness of the instrument. Please explain. **Answer**: Vivian Sullivan stated, "The QA checks are the item addressed in #16." However, question 16 (above) addresses QC checks. The laboratory needs to understand the difference and functions of QA and QC.
18	Appendix A,	**EPA**: The text states, "Prepare the sample and place it on the detector for counting" but does not

	section 2.1.1	provide information on how the sample is prepared. Please provide operational details on sample preparation. **Answer**: The laboratory SOP uses "may"; optional language is not appropriate for an SOP because the SOP must state what is done and how, not what may be done.
19	Appendix A, section 3.3	**EPA**: The text states, "Create an analysis worksheet for each sample." If this worksheet was created by the laboratory, please provide validation records for review. **Answer**: ACL did not provide an answer.
20	Appendix A, section 3.4	**EPA**: The text states, "Create a table in the summary data sheet for data reporting. Batch calculations, such as switching the units from per mL to total activity may be performed on this sheet." If this worksheet was created by the laboratory, please provide validation records for review. **Answer**: ACL did not provide an answer.
21	Appendix B, section 1.2.1	**EPA**: The text states, "Standards for quality assurance checks will be run using NIST traceable standards. Run the efficiency protocol with both an alpha and a beta standard for each detector used. Check the efficiency through the data output function. Save the data to the control chart. If the system has been idle for a while, save as the official value. Check the control chart to confirm efficiency is stable for both alpha and beta." Please explain the underlined text and what "the official value" is. **Answer**: ACL provided an adequate response to this question.
22	Appendix B, section 1.2.3	**EPA**: The text states, "Control charts are maintained by the software, but often do not print in a readable format. An Excel table of the control check pass/fail and background pass/fail may be made." Please explain how the laboratory verifies that this software is functioning correctly and the pass/fail limits used and their origin. **Answer**: ACL responded to this question but it is not clear from the response that the appropriate verifications are performed.
23	Appendix B, section 3.1	**EPA**: The text states, "Use the alpha beta Excel template to record the tare and gross weights of the planchets." If this worksheet was created by the laboratory, please provide validation records for review. **Answer**: ACL stated that the Excel spreadsheet was checked when the data package was reviewed.
24	Appendix C,	**EPA**: Appendix C, section 1.1.1, states, "Run the background protocol, with empty cells, for all

	section 1.1.1	detectors to be used. Check the QC report for any cells that do not pass the background tests (all isotopes detected above limits will be listed). Cells that fail will be locked out." Please provide the pass/fail acceptance criteria used by the laboratory unless these are assigned by the instrument software. **Answer**: The criteria are assigned by the instrument software. This is adequate.
25	Appendix C, section 1.2.1	**EPA**: The text states, "Standards for quality assurance checks will be run using multi-element NIST traceable standards. Run the QC check protocol with the standards for each detector used. Check the QC report for any cells that do not pass the QC check. Cells that fail will be locked out." Please provide the pass/fail acceptance criteria used by the laboratory unless these are assigned by the instrument software (see 27, below). **Answer**: The criteria are assigned by the instrument software. This is adequate.
26	Appendix C, section 1.2.3	**EPA**: Please provide the acceptance criteria for the "QC and pulser counts" unless these are assigned by the instrument software. **Answer**: The criteria are assigned by the instrument software. This is adequate.
27	Appendix C, section 1.2.4	**EPA**: The text states, "Alpha analyst prints reports for all cells for each check. Any deviation will be reported in detail in the report." Please explain what the laboratory defines as a "deviation" and if/how the corrective action process is used to resolve them. **Answer**: ACL personnel interviewed stated that what was really meant by "deviation" was "flags." ACL needs to revise the SOP to reflect what is done and to clarify the language.
28	Appendix C, section 1.2.4	**EPA**: The text states, "<u>If specific peaks are not well resolved, the sum of those peaks may be used in the data analysis in comparison with gamma and ICP-MS data</u>. The peaks are not well resolved if the analyst cannot set an ROI around each peak that approaches the baseline of the spectra." Please explain the genesis and technical basis for the process underlined. If this was developed by the laboratory, please provide the validation data for review. **Answer**: Vivian Sullivan stated that this method was developed by the laboratory. "We do not have a specific validation set, it is a process developed over time, only performed by analysts with experience in the technique."
29	General	**EPA**: Please provide a comprehensive list of all radiometric equipment used to generate data in the laboratory (or for BDR 7TRUA50-A), including: a. Name and identifier of the instrument. b. Date of initial calibration.

		c. Comprehensive listing of all calibrations or instrument response characterizations for the instrument, e.g., FWHM, energy/channel, efficiency/energy gamma spectrometers. d. Written procedure(s) that control the instrument's calibration(s), maintenance, performance checks and use. e. Location of the documentation for the instrument's calibration and performance checks. f. Formal measurement control program that encompasses the instrument. **Answer**: See Attachment A, Table A-2, General Questions, Nos. 3, 7, 9, 10 and 11. a. Vivian Sullivan provided a list. b. Vivian Sullivan provided a list. c. Not included in list. d. Vivian Sullivan provided list of procedures but they do not address all aspects. e. Instrument calibrations and performance checks are documented in logbooks and BDRs. f. ACL does not have a formal measurement control program.
30	General	**EPA**: Please provide a comprehensive list of all radionuclide standards/source that are or have been used for instrument calibration, including the following for each standard/source: a. Radionuclide, physical form and activity as of a specified reference date. b. Pedigree of standard/source (traceable to the national standard base). c. Specific standard/source emissions used for calibration and applicable analytical methods (ACL SOPs), e.g., specific photon emission lines. d. Documentation for a. through c. **Answer**: Vivian Sullivan provided a list to EPA; see Table A-2, General Questions, No. 3.
Supplemental Quality Assurance Project Plan, 10-0059		
1	General	**EPA**: This document does not appear to be dated, approved or controlled. Please explain. **Answer**: ACL provided a signed and dated copy of the document. This is adequate.
2	Section 2.1	**EPA**: The text states, "The sample is prepared according to the client work plan, with observation by ACL personnel if requested in the work plan." The Supplemental Quality Assurance Project Plan, 10-0059, does not include any sample preparation instructions and this text says that ACL personnel do not perform sample preparation. Please explain, clarify and verify who prepared the samples reported in BDR 7TRUA50-A. **Answer**: ICP-MS personnel interviewed stated that they did not receive any information about any special requirements for the WIPP samples. Although the QA/QC coordinator stated that they

		did have training on the requirements, no records were kept of the training topics, when training was held and who attended.

[a] EPA derived answers to its evaluation questions by interviewing ACL personnel and observing laboratory records during its July 31–August 1, 2012, evaluation of ACL.

Table A-2. EPA Evaluation Questions – Radiometric Equipment and Documentation

Question No.	EPA Questions and Answers Derived from EPA Evaluation July 31–August 1, 2012[a]
General Questions	
1	**EPA Question (EPA):** What is the source of all alpha & gamma energy, transition probability and half-life values? **Answer Derived from EPA Evaluation (Answer):** Values are based on LBNL data; Vivian Sullivan provided an Excel spreadsheet containing all relevant values.
2	**EPA:** Obtain a copy of the gamma library *CHTRU A.Lib*. **Answer:** ACL provided this gamma library.
3	**EPA:** Identify the SOPS for instrument maintenance/control, including applicable revisions. **Answer**: There is no SOP for instrument maintenance/control.
4	**EPA**: Obtain comprehensive list of all radionuclide standards/sources used for instrument calibration & performance testing, including: radionuclide; radiation of interest; activity as of reference date; impurities; traceability to national standards base; useable life; and current status; name of commercial vendor supplying standard/source. **Answer**: Vivian Sullivan provided a list.
5	**EPA**: Identify means of decay correcting standards/sources listed above. **Answer**: Decay corrections are instrument specific, i.e., performed by the software associated with each radiometric instrument (alpha and gamma spectrometers); performed manually for Tennelec radionuclides, as needed.
6	**EPA**: Obtain the equivalent of an LOQI for laboratory/counting room personnel. **Answer**: ACL training records do not address all aspects of relevant training directly.
7	**EPA**: Identify the general procedures/protocols for taking an instrument out of service, lock-out/tag-out, etc. **Answer**: There is no ACL SOP or formal procedure for these actions.
8	**EPA**: Describe the recent renovation of the counting room and its impact: were instruments moved, taken out of service, were calibrations checked/verified post-renovation? **Answer**: Instruments that were moved were brought back into service; calibrations are supposed to be checked prior to measuring samples according to SOPs.
9	**EPA**: Is there a formal program/SOP/written instruction for documentation or control of laboratory notebooks? **Answer:** No.
10	**EPA**: Identify the SOPs or other written instructions that address instrument calibration, operation and maintenance.

	Answer: Calibration and maintenance are not addressed in any SOP. Instrument operation is addressed in a cursory manner in method-specific SOPs; e.g., SOP:ACL-084 addresses operation of the gamma systems.
11	**EPA**: Is there a formal software control program for radiometric equipment at the ACL (NQA-2.7)? **Answer:** There is no formal program of this type at ACL.
Tennelec GPC	
1	**EPA**: How many detectors does the system have? **Answer**: Eight.
2	**EPA**: How old is the system? **Answer**: The system dates to the late 1990s.
3	**EPA**: Does the system use P-10 gas? **Answer**: Yes, supplied by manifold.
4	**EPA**: Is there a protocol for changing gas tank and performance testing post-change? **Answer**: No.
5	**EPA**: Are all detectors operational? **Answer**: Yes.
6	**EPA**: Is there documentation of the system's maintenance/performance history? **Answer**: The Tennelec's maintenance and performance history is addressed in ACL logbooks.
7	**EPA**: Do the systems usually operate at the "Combined Voltage"? **Answer**: Yes.
8	**EPA**: Are any data obtained at "Alpha Only" voltage? **Answer**: No.
9	**EPA**: What are the operating voltages of the Tennelec, "Combined" and "Alpha Only"? **Answer**: 1,482 volts for "Combined;" "Alpha Only" was not specified.
10	**EPA**: When was the operating voltage (Plateau Curve) determined initially and most recently? **Answer**: The initial determination is unclear; the most recent determination is listed as 2008 in the logbook.
11	**EPA**: What are values for crosstalk: alpha into beta and beta into alpha? **Answer**: The value for the alpha into beta ratio is 0.08.

12	**EPA**: What radionuclides were used for the crosstalk determination? **Answer**: ^{90}Sr for beta and ^{241}Am for alpha.
13	**EPA**: Do they measure U_{Nat}? [^{214}Bi $\beta = 3.26$ MeV E_{max}; ^{90}Y $\beta = 2.27$ MeV; ^{90}Sr $\beta = 0.56$ MeV] **Answer**: U is measured; Vivian Sullivan was not aware of the higher crosstalk potential of ^{214}Bi relative to ^{90}Y.
14	**EPA**: When were these determined initially and have most recently? **Answer**: Initially performed in 1999, most recent appears to be 2008.
15	**EPA**: What is the thickness of the Tennelec windows? **Answer**: Vivian Sullivan did not know; Tennelec says 80 micrograms per gram is standard window thickness.
16	**EPA**: Have any of the Tennelec windows been replaced? If so, how many and when? **Answer**: There are no records of any replacements.
17	**EPA**: When was the absolute efficiency (cpm/dpm) determined for alpha and beta? **Answer**: ACL has done this "intermittently" since the late 1990s. The last time appears to be 2 years ago (the records are not clear); ^{241}Am was used for alpha and ^{90}Sr was used for beta.
18	**EPA**: Has this changed since initial determination? **Answer**: It appears so; the records are not clear.
19	**EPA**: When was the self-absorption (Transmission Factor) curve produced initially? **Answer**: The self-absorption curve was generated once, in the 1990s; no records of a subsequent curve are available.
20	**EPA**: What are the upper mass (density) limits for alpha & beta? **Answer**: 100 mg (~5 mg/cm^2).
21	**EPA**: Has this changed since the initial determination? **Answer**: No.
22	**EPA**: What radionuclides are used for alpha & beta routine performance checks? **Answer**: ^{90}Sr for beta and ^{241}Am for alpha.
Gamma Detectors	
1	**EPA**: How many detectors does the system have? **Answer**: There are four operational gamma detectors that can be used for WIPP samples.
2	**EPA**: How old are the detectors? **Answer**: The oldest detector is from the mid-1990s.

3	**EPA**: Are all detectors operational? **Answer**: Yes.
4	**EPA**: Is there documentation of the system's (detectors') maintenance/performance history? **Answer**: Some aspects of the detectors' performance history are documented.
5	**EPA**: Identify the name and version of data acquisition and analysis software. **Answer**: ACL provided specifications for all four gamma detectors.
6	**EPA**: Identify the detectors' type and efficiencies. **Answer**: See previous answer.
7	**EPA**: When was the absolute efficiency (cpm/dpm) determined? **Answer:** Not clear based on documentation.
8	**EPA**: What radionuclide(s) was used for this determination? **Answer**: Not clear based on documentation.
9	**EPA**: What radionuclide(s) is used for routine performance checks? **Answer**: ^{154}Eu and ^{137}Cs.
10	**EPA**: Has this changed since the initial determination? **Answer**: Not clear based on documentation.
11	**EPA**: It appears a (rate loss) pulser is not used for the gamma system. **Answer**: A pulser is not used.
12	**EPA**: Is there a formal listing of all calibrated geometries? **Answer**: No listing was provided.
13	**EPA**: Is there a separate efficiency, energy and resolution calibration for each detector for each geometry? **Answer**: Yes.
14	**EPA**: Is there a specific criterion for excess dead-time? **Answer**: No specific criterion, ~30% is used as a rough guide.
15	**EPA**: Are filters (Cd, Sn, Al) ever used for high dead-time samples? **Answer**: Filters are not used.
16	**EPA**: Why is the 414-keV ^{239}Pu line not shown (equivalent to the 375- and 129-keV lines)? **Answer**: Question not addressed.

17	**EPA**: Details of correction 662-keV ROI for ^{241}Am to provide ^{137}Cs when ^{241}Am is detected? **Answer**: ACL uses the correction built into the analysis software.
Alpha Spectrometers	
1	**EPA**: How many detectors does the system have? **Answer**: 24 detectors total; 14 are used for WIPP.
2	**EPA**: Are all detectors operational? **Answer**: Yes.
3	**EPA**: How old are the detectors? **Answer**: Ten non-WIPP detectors were obtained in 2011; 14 WIPP detectors were obtained in 2000.
4	**EPA**: Is there documentation of the system's (detectors') maintenance performance history? **Answer**: There is a logbook for operation; there is not a logbook for maintenance.
5	**EPA**: Identify the name and version of data acquisition and analysis software? **Answer**: Verified during evaluation.
6	**EPA**: When was the absolute efficiency (cpm/dpm) determined? **Answer**: Appears to have been initially determined in 2000 for older detectors; performed most recently for new detectors in 2011; both values are listed as 0.2 cpm/dpm irrespective of energy.
7	**EPA**: What radionuclide(s) was used for this determination? **Answer**: ^{238}U, ^{239}Pu and ^{243}Cm were used; a general value of 0.2 cpm/dpm is used.
8	**EPA**: What radionuclide(s) is used for routine performance checks? **Answer**: ^{238}U, ^{239}Pu and ^{243}Cm are used.
9	**EPA**: Has this changed since the initial determination? **Answer**: No.
10	**EPA**: Is this geometry/sample configuration specific? **Answer**: Yes, the geometry is a disk at ~2 cm from the detector.
11	**EPA**: Has this changed since the initial determination? **Answer**: Unclear.
12	**EPA**: How are ROIs set? **Answer**: ROIs are based on operator's judgment.

13	**EPA**: Is there a formal calibration and acceptance criterion for resolution (FWHM)? **Answer**: No, the goal is derived based on a percent of the total activity.
14	**EPA**: It appears a pulser is not used for the alpha spectrometers. The BDR for SDG: 7TRUA50-A showed a pulser at 5 MeV. **Answer**: Vivian Sullivan stated that a pulser should have been used but mistakenly was omitted from BDR SDG: TIC-1 and the oversight was not caught during subsequent reviews.
15	**EPA**: Establish the specifics of sample mountings; is there more than one option? **Answer**: Direct deposition on a planchet is only option.
16	**EPA**: Have they ever had a recoil problem? **Answer**: Not to date.
17	**EPA**: Were any alpha detectors replaced due to contamination? If so, which ones and when? **Answer**: None have been replaced due to contamination to date.
18	**EPA**: Is there a criterion for gross or radionuclide-specific activity in counting chamber? **Answer**: The criterion used is the default value in the Canberra Analyst software.

[a] EPA derived answers to its evaluation questions by interviewing ACL personnel and observing laboratory records during its July 31–August 1, 2012, evaluation of ACL.

ATTACHMENT B

LIST OF DOCUMENTS REVIEWED BY EPA DURING EVALUATION

10-0059 Training Records for Del Bowers, Michael Kalensky, Vivian Sullivan, and Yifen Tsai, various dates

Questions for Argonne National Laboratory-East Analytical Chemistry Laboratories (ANLE-ACL) EPA evaluation July 31–August 1, 2012

ACL Answer to EPA Questions #1–3 for BDR 7TRUA50-A, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #4 for BDR 7TRUA50-A, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #5 for BDR 7TRUA50-A, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Questions #1 and 2 for NCR-RHANL-2344-11, undated, prepared for EPA evaluation July 31, 2012

ACL Answers to EPA Questions for SOP ACL-271, undated, prepared for EPA evaluation July 31, 2012

ACL Answers to EPA Questions #1–18, 21–28 for SOP ACL-274, undated, prepared for EPA evaluation July 31, 2012

ACL Answers to EPA Questions #19 and 20 for SOP ACL-274, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #29 for SOP ACL-274, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #30 for SOP ACL-274, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #1 for S-QAPP 10-0059, undated, prepared for EPA evaluation July 31, 2012

ACL Answer to EPA Question #2 for S-QAPP 10-0059, undated, prepared for EPA evaluation July 31, 2012

Analytical Chemistry Laboratory ACL Notebook Inventory – Group 1, Revised March 2012

Analytical Chemistry Laboratory ACL Notebook Inventory – Group 2, Revised September 2011

Analytical Chemistry Laboratory ACL Notebook Inventory – Group 3, Revised December 2010

Analytical Chemistry Laboratory Reporting Analytical Results, Revision 10, undated

BDR 7TRUA50-A; Analytical Chemistry Laboratory Report of Analytical Results for ACL Nuclear Operations Division, 205 K-Wing, SDG:7TRUA50-A (ACL: 10-0059A)

BDR 7TRUA50-A, Certificates – Alpha, Appendix C, p. 7, 9–11, 16–18

BDR 7TRUA50-A, Certificates – Alpha/Beta, Appendix B, p. 3–4, 7–12

BDR 7TRUA50-A, Certificates – Gamma, Appendix A, p. 5–15

BDR 7TRUA50-A, Certificates – ICP-MS, Appendix D, p. 2–3

BDR 7TRUA50-A, QA Charts – Alpha, Appendix C, p. 2–5, 19–28

BDR 7TRUA50-A, QA Charts – Alpha/Beta, Appendix B, p. 2

BDR 7TRUA50-A, QA Charts – Gamma, Appendix A, p. 2–3

BDR 7TRUA50-A, QA Charts – ICP-MS, Appendix D, p. 4, 9, 18, 29, 31, 35, 38, 43, 51 and 61

BDR TIC-1; Analytical Chemistry Laboratory Report of Analytical Results for ACL Nuclear Operations Division, 205 K-Wing, SDG:TIC-1 (ACL: 11-0197)

BDR TIC-1, Certificates – Alpha, Appendix C, p. 9–11, 16–17

BDR TIC-1, Certificates – Alpha/Beta, Appendix B, p. 35–38, 41–46

BDR TIC-1, Certificates – ICP-MS, Appendix D, p. 26

BDR TIC-1, Certificates – Gamma, Appendix A, p. 22–25

BDR TIC-1, QA Charts – Alpha, Appendix C, p. 3–5

BDR RIC-1, QA Charts – Alpha/Beta, Appendix B, p. 3–33

BDR TIC-1, QA Charts – ICP-MS, QA Charts – ICP-MS, Appendix D, p. 9–10, 12 and 25

Calibration Certificate 2010, Balances, Serial Nos. 23750304, 1128242333, H34681 and 23750303, January 26, 2010

Calibration Certificate 2011, Balances, Serial Nos. 23750304, 23750303, 1128242333 and H34681, January 28, 2011

Calibration Certificate 2012, Balances, Serial Nos. 32513, 1128242333, H34681, 23750304 and 23750303

Documents Related to the Mixed Analyte Performance Evaluation Program (MAPEP), various dates

SOP ACL-070, Standard Operating Procedure: Quality Assurance, Calibration and Counting Protocols for Gamma Spectra of Samples, Revision 01, October 17, 2007

SOP ACL-084, Standard Operating Procedure: Analysis and Calculation of Gamma Radioactivity, Revision 01, October 17, 2007

SOP ACL-095, Standard Operating Procedure: Gross Alpha and Beta Radioactivity, Revision 04, October 17, 2006

SOP ACL-166, Standard Operating Procedure: Implementation and Documentation of Corrective Actions, Revision 05, August 31, 2009

SOP ACL-179, Standard Operating Procedure: Measurement and Test Equipment (Balances and Automatic Pipets) Inventory and Operational Verification, Revision 02, January 18, 2008

SOP ACL-258, Standard Operating Procedure: Alpha Scan for Actinides Utilizing Extraction Chromatography, Revision 00, October 17, 2007

SOP ACL-259, Standard Operating Procedure: Obtaining Gamma Spectra for Samples in a Calibrated Geometry Using the Ortee GammaVision Program, Revision 00, October 17, 2007

SOP ACL-262, Standard Operating Procedure: Data Verification and Validation, Revision 00, October 10, 2008

SOP ACL-271, Standard Operating Procedure: Determination of Trace Elements/Isotopes in Solution by Inductively Coupled Plasma Mass Spectrometry (ICPMS) Using the PerkinElmer SCIEX ELAN DRC II, Revision 00, August 1, 2011

SOP ACL-274, Standard Operating Procedure: Isotopic Analysis of Samples for Disposal at WIPP, Revision 00, January 14, 2010, Revision 01, March 15, 2010, Revision 02, July 14, 2010

SOP ACL-280, Standard Operating Procedure: Gross Alpha and Beta Radioactivity Determination Using a Gas Proportional Counter, Revision 00, January 19, 2012

SOP ACL-281, Standard Operating Procedure: Sample Preparation and Measurement for Gamma Spectrometry, Revision 00, January 19, 2012

SOP ACL-282, Standard Operating Procedure: Sample Preparation for Radioactive Analysis, Revision 00, January 19, 2012

Technical Basis, Report of Analysis of Smear Data for Alpha Gamma Hot Cell Facility, undated

ATTACHMENT C-1

EPA INSPECTION ISSUE TRACKING FORM, ISSUE NO. ANL-CCP-RH-ACL-2012-01CR, DRAFT

Inspection No. ANL-ACL-Laboratory-2012	**Issue Number:** ANL-CCP-RH-ACL-2012-01CR **Date:** August 1, 2012
Inspector: D. Gill & P. Kelly **Attachments?** ☐ YES ☒ NO	**Sample Size:** NA **Population size (if known):** NA
Description of Issue: Based on EPA's sample, the ACL was not adequate in the following fundamental technical areas: measurement control; records; training; standard operating procedures (SOPs); and communication of WIPP-specific requirements to analysts. Please see Section E, below.	
B. Regulatory Reference: 40 CFR 194.24	
C. Site requirement(s): Not applicable	
D. Discussed with: M. Pinzel, I. Quintana, V. Sullivan, D. Pancake, & D. Dietzel	
E. Additional Comments: Prior to the generation of any data that will be used to support WIPP characterization by the ACL, the laboratory's operations must be formally evaluated and approved by EPA.	
F. Site Response Information: **Site Response Required?** ☒ YES ☐ NO **Site Response Due Date:** Contingent upon laboratory funding	

ATTACHMENT C-2

EPA INSPECTION ISSUE TRACKING FORM, ISSUE NO. ANL-CCP-RH-ACL-2012-02C, DRAFT

Inspection No. ANL-ACL-Laboratory-2012	**Issue Number:** ANL-CCP-RH-ACL-2012-02C **Date:** August 1, 2012
Inspector: D. Gill & P. Kelly **Attachments?** ☐ YES ☒ NO	**Sample Size:** NA **Population size (if known):** NA
Description of Issue: Based on EPA's sample, the ACL was not adequate in the following fundamental technical areas: measurement control; records; training; standard operating procedures (SOPs); and communication of WIPP-specific requirements to analysts.	
B. Regulatory Reference: 40 CFR 194.24	
C. Site requirement(s): Not applicable	
D. Discussed with: M. Pinzel, I. Quintana, V. Sullivan, D. Pancake, & D. Dietzel	
E. Additional Comments: NA	
F. Site Response Information: **Site Response Required?** ☐ YES ☒ NO **Site Response Due Date:** NA	