

DOE Laboratory Accreditation Program

**OVERVIEW OF THE DOELAP FOR  
RADIOBIOASSAY OVERSIGHT BOARD**

# DOELAP OVERSIGHT BOARD MEMBERS

- × Eugene Carbaugh (Hanford)
- × David Hickman (LLNL)
- × Jay MacLellan (Hanford)
- × Charles Potter (Sandia)
- × Govind Rao (ORNL)

# OB AUTHORITY PER DOE-STD-1111-98

- ✘ The OB is an advisory body
- ✘ Decisions and recommendations are non-binding on the DOELAP Administrator
- ✘ Decisions and recommendations carry significant weight on the conduct of DOELAP

# OB ACCREDITATION REVIEW PROCESS

- ✘ Establish a quorum (3 members)
- ✘ Voting criteria by a simple majority of all members (i.e., 3 members)
- ✘ Excused from evaluation and voting when a conflict of interest exists (a member will be recused when the member's site program has a petition before the Board)

# NOTES REGARDING SUBSEQUENT SLIDES

- ✘ DOE-STD-1111-2012 has not been released.
- ✘ Changes to DOE-STD-1111-2012 may not be reflected in the subsequent information.
- ✘ Use the following information from DOE-STD-1111-2012 with caution.

## **DOE-STD-1111-98**

- ✘ Support the DOELAP Administrator to maintain long term continuity of radiobioassay programs
- ✘ Ensure technical quality and consistency of DOELAP technical standards and site assessments

## **DOE-STD-1111-2012**

Removed

## DOE-STD-1111-98

- × Review PEPA recommendations
- × Review corrective action plans for concerns or deficiencies
- × Advise DOELAP Admin. regarding approval or denial
- × Recommend appropriate changes to DOELAP Admin.
- × Evaluate PTL “biannually”

## DOE-STD-1111-2012

- × Review (STM) recommendations and referred requests for modification
- × Advise the DOELAP Admin. on further action re: recommendations and referred requests
- × Prepare record of meetings and archive with STM
- × Advise DOELAP Admin. on issues pertinent to DOELAP
- × Assess PTL biennially

# OB RESPONSIBILITIES

# DOE-STD-1111-2012 NEW CONTENT PERTINENT TO THE OB

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- ✘ A new external dosimetry or radiobioassay program that has been accredited shall be assessed approximately one year after the effective date. Continuation of the accreditation period may be granted following an onsite assessment and an Oversight Board's recommendation based on the assessment.
- ✘ An amendment request to add one or more performance categories shall require performance testing in those categories and an Oversight Board review. This type of amendment shall be coordinated with the appropriate STM.

The OB looks for a completed accreditation package.

The OB looks at each element in the accreditation package.

The accreditation package must in-and-of itself justify the recommendations provided to the DOLEAP Administrator

- ✘ Application & DOE Site Office concurrence
- ✘ Performance test results
- ✘ Assessment
- ✘ Corrective Action Plan (CAP)
- ✘ PEPA (STM) recommendation

## **ELEMENTS OF THE OB REVIEW**

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## Application & DOE Site Office Approval

Example of what OB does not like on an application:

10. Describe the efficiency calibration and routine counting procedures in the indirect radiobioassay measurement.

ANS. "GPC counting use a self absorption curve"

Other examples are included in your handout.

- ✘ Electronic submission ensures DOE Site approval
- ✘ Are all sections properly completed?
- ✘ For each process and measurement system to be accredited, have all of the listed elements in the application questions been addressed?
- ✘ Does the application describe the processes, methods, and measurement systems that were performance tested?
- ✘ Do the application answers demonstrate that the applicant is knowledgeable about sample processing (including the radiochemistry), measurements and data computations (especially when using a Vendor laboratory)?

**ELEMENTS OF OB REVIEW (CONTINUED)**

## Performance Test Results

- ✘ Are the results within the performance requirements given in DOE-STD-1112?
- ✘ Do the PTL test samples meet the requirements in DOE-STD-1112 (e.g., MTL)....was it an appropriate test?
- ✘ Some interest in large variations and results that barely pass.
- ✘ If Failures – get information from PEPA, application, assessor report, and CAP to determine if there is an explanation for the failure.

**ELEMENTS OF OB REVIEW (CONTINUED)**

# Assessor Report

Assessors are the eyes & ears of the OB

## Things that help the OB:

Use descriptive sections or observations (if not a concern) that describe processes involved especially for failed performance test result(s).

(e.g., OBS: A direct aliquot analysis method with an open LSC energy window is used for H-3 since the normal sample for this site is expected to only contain H-3. The PT sample contained both H-3 and C-14.)

- ✘ Did the assessment review the processes, methods, and measurement systems listed in the application?
- ✘ Were there deficiencies and concerns from the assessment?
- ✘ Were past concerns addressed?
- ✘ For New Deficiencies or Concerns, are they consistent with the intent of DOE-STD-1112?
- ✘ Are there observations or concerns that help to explain issues observed in the performance tests?
- ✘ Noteworthy practices can have an influence!

## ELEMENTS OF OB REVIEW (CONTINUED)

## Corrective Action Plan

- ✘ Was CAP submitted through the DOE Site Office?
- ✘ Were all deficiencies and concerns addressed?
- ✘ Did the Assessors review the CAP before submittal to the OB? (What is their response to the CAP?)
- ✘ Do the responses to Deficiencies and Concerns appear to address the issue and is the corrective action reasonable?
- ✘ Did the applicant fail to adequately respond or address a deficiency or concern? (bad news)
- ✘ Does the attitude within the verbiage of the CAP reflect a constructive desire to improve? (not a game buster, but can influence the overall impression of the OB towards the applicant)

**ELEMENTS OF OB REVIEW (CONTINUED)**

## PEPA (STM) Recommendation

- ✘ YES or NO
- ✘ If No, then the OB is very likely to also be a NO!
- ✘ By the time an accreditation package reaches the OB, the PEPA should be an advocate for the applicant!

**ELEMENTS OF OB REVIEW (CONTINUED)**

## After all Package Reviews

- Completed before the OB adjourns

- ✘ Finalize the recommendations
- ✘ Generate recommendation letters to DOELAP Administrator
- ✘ Complete meeting minutes

**ELEMENTS OF OB REVIEW (CONTINUED)**

# ADDITIONAL OB ACTIVITIES

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- ✘ Review PTL activities over the past year since the previous OB meeting.
- ✘ Assess the PTL biennially for conformance with applicable procedures, standards, and accreditations.
- ✘ Technical Equivalency reviews (these can take 3 – 4 weeks if performed outside the annual OB meeting)

# **AN IMPORTANT PARAGRAPH IN THE OB RECOMMENDATIONS TO THE DOELAP ADMIN.**

**“It should be noted that completion of corrective actions to resolve findings identified during the site assessment is necessary to be in full compliance with the requirements of the DOE Technical Standards pertinent to DOELAP. The Board requests that the DOE Field Office ensure that the response plan is followed.”**

## Assessor Observations

- ✘ Observations are used for a variety of inputs from assessors (noteworthy and CQI)
- ✘ Some sites have a requirement to enter all observations from external assessments into a corporate system that requires causal analysis, identification of corrective action, and follow-up.
- ✘ Some on the OB felt that observations should be limited to marginal items or items where implementation would provide a large quality improvement
- ✘ The OB recommended to the PEPA this issue be discussed at assessor training in 2012.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS**

## When is Technical Equivalency Needed?

DOE-STD-1111-2012: A change to a primary component of an accredited or qualified external dosimetry or radiobioassay system, or a change in the administrative structure (as described in the application) of an accredited external dosimetry or radiobioassay program, shall be authorized by a determination of technical equivalence prior to the change going into effect. A request for technical equivalence should be received by the STM no less than 45 calendar days before the proposed date of the change for issuance of a timely response.

- ✘ Changes to methods & systems (with acceptance criteria) need to be documented by the site.
- ✘ Establishing more formal guidance regarding TE would be useful. Use the PEPA for now.
- ✘ Repair or replacement of like equipment should not need TE if adequately covered by routine procedures and QC practices (e.g., Same model # PIPs detector replacement).
- ✘ Simply expanding an existing system (e.g., additional detectors to an alpha system) would not require TE.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS (CONT.)**

## DOE site laboratories that provide services to other DOE site laboratories:

DOE-STD-1111-2012 provides a Vendor Qualification that will negate these OB concerns and will establish a new process.

- ✘ Vendor qualification is not limited strictly to commercial laboratories, but extends to DOE site labs that perform analytical services for multiple sites.
- ✘ Such DOE labs shall undergo annual performance testing as do the commercial labs under the proposed vendor qualification program.
- ✘ Accredited sites must perform annual QA assessments of non-site service labs.
- ✘ Rationale: the failure of a lab providing services to multiple sites potentially affects all sites using the lab. Requiring annual testing of such service labs is a safe and logical approach to treat vendors alike.
- ✘ DOELAP service providers providing service to other DOE sites, (e.g., GEL, Test America Laboratories , and ORNL) undergo annual performance and triennial onsite assessments.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS (CONT.)**

## Clinical Laboratory Improvement Amendments (CLIA)

- ✘ The PEPA presented an issue to the OB regarding a state regulator requiring CLIA certification on a potential candidate DOELAP service provider.
- ✘ The DOELAP administrator wrote a memo indicating the CLIA is not applicable to DOELAP.
- ✘ There is some concern that other state regulators might take similar actions.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS (CONT.)**

## Refractory plutonium

- ✘ Performance testing does not use refractory plutonium in the artificial fecal matrix.
- ✘ This raises some questions about the adequacy of site procedures for dissolution of worker samples containing refractory plutonium.
- ✘ The OB recommends that the PTL explore the possibility of including refractory plutonium in a fecal matrix.
- ✘ A voluntary test round with site labs is recommended prior to actual performance testing using any new matrix.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS (CONT.)**

# Limited Accreditation for short-term DOE Contractors/Projects

(facilities or projects of either short duration or needing a limited amount of analysis - two-to-three years & less than the length of the accreditation cycle - or less than or equal to 50 monitored personnel per year)

- ✘ The applicant would be required to demonstrate familiarity with the services provided by the DOELAP-accredited laboratory.
- ✘ The PEP A and Performance Administrator should determine that the exception process as defined in section 7 of DOE-STD-III-98 is not applicable.
- ✘ The facility or project would provide a justification for limited accreditation and identify the DOELAP accredited bioassay service provider with their application.
- ✘ Performance testing and assessment of the DOELAP accredited provider need not be performed
- ✘ Under the recommendation of the PEP A, an onsite assessment limited to a review of the personnel and limited quality program could be performed.
- ✘ The PEP A would review the application for completeness and prepare a recommendation to the OB.
- ✘ The OB would review and vote on the package by email or at the next meeting if possible and prepare a recommendation for the Program Administrator who would make the final determination.

**INTERESTING DISCUSSIONS OF THE OB OVER THE PAST FEW YEARS (CONT.)**

# HANDOUTS – GOOD, BAD, AND UGLY APPLICATION SUBMISSIONS

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## × Direct

- + Good Application
- + Bad Application
- + Too Long Application

## × Indirect

- + Good Application
- + Bad Application

“The Oversight Board recommends that accreditation be contingent upon Laboratory X properly completing the DOELAP application (e.g., System, Counting Configuration, Data Reduction Algorithms, Calibration, etc.) and submission to the PEPA for final retention.”

## WELL COMPLETED PART OF A DIRECT RADIOBIOASSAY APPLICATION

Answers to items 7-10 (see previous page for measurement categories)

Item 7:

[REDACTED] has two physical counters. First, the ACCUSCAN-II Scanning Whole-Body Counter is used for the measurement of Category V (Mn-54, Co-58, Co-60 – lungs) and Category VI (whole body) radionuclides. Second, a portable High Purity Germanium (HPGe) detector system is used for the measurement of Category VII radionuclides in the Thyroid.

Item 8:

Radionuclides Categories V and VI:

The ACCUSCAN-II scanning whole body counter is used for the analysis of the marked nuclides in categories V and VI. The system consists of two P-type HPGe Canberra detectors, each with a relative efficiency of 40% and a 1.5 – 2 inch thickness of copper-lined lead shielding surrounding the detectors. The shielding consists of a minimum 4-inch thick steel shadow shield. The subject vertically stands within the shadow shield and is counted for 600 seconds. The Canberra ABACOS software is employed to collect the spectrum and analyze it. The collected spectrum is analyzed by performing a peak search, peak area calculation, background correction, computing the counting efficiency for each found peak, isotope identification and computing MDAs for each radionuclide, and generating a report. The system is energy calibrated using a mixed gamma source, which has an energy range from 88 keV to 1836 keV (with an optional energy line at 59 keV). The Canberra RMC-II phantom is used for efficiency calibrations. There are four types of efficiency calibrations that can be performed using the RMC-II phantom (1) GI Tract, (2) Thyroid, (3) Lung, and (4) Whole Body geometry. For a routine whole body count the whole body geometry calibration is used due to this geometry being the most conservative. A blank resin filled BOMAB is counted periodically and used for background correction. Typical MDA values are 5 nCi for Co-60 and 6 nCi for Cs-137.

Radionuclides Category VII:

A portable HPGe detector with approximately a 30% relative efficiency is used for counting radionuclides in the thyroid category. This detector is shielded with lead and may be n-type/or p-type. The detector is placed over the subject's thyroid and counted for 60 seconds. The signals from the detector are sent to an inspector unit, which houses the NIM electronics. The Canberra GENIE software is employed to collect the spectrum and analyze it. The system is energy calibrated using a mixed gamma source. A 5ml glass ampoule and/or a 30ml plastic vial source is used in conjunction with the ANSI N13.30 cylindrical thyroid phantom to generate an efficiency versus energy curve. The analysis consists of a peak search, peak area calculation, background correction, counting efficiency computation for each peak, radionuclide identification and interference correction, MDA computation for each radionuclide, and report generation. A typical MDA for I-131 is 2.5 nCi.

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### Radionuclides Category VI:

8. The ACCUSCAN-II scanning whole body counter is used for the analysis of the marked nuclides in categories V and VI. The system consists of two P-type HPGe Canberra detectors, each with a relative efficiency of 40% and a 1.5 – 2 inch thickness of copper-lined lead shielding surrounding the detectors. The shielding consists of a minimum 4-inch thick steel shadow shield. The subject vertically stands within the shadow shield and is counted for 600 seconds. The Canberra ABACOS software is employed to collect the spectrum and analyze it. The collected spectrum is analyzed by performing a peak search, peak area calculation, background correction, computing the counting efficiency for each found peak, isotope identification and computing MDAs for each radionuclide, and generating a report. The system is energy calibrated using a mixed gamma source, which has an energy range from 88 keV to 1836 keV (with an optional energy line at 59 keV). The Canberra RMC-II phantom is used for efficiency calibrations. There are four types of efficiency calibrations that can be performed using the RMC-II phantom (1) GI Tract, (2) Thyroid, (3) Lung, and (4) Whole Body geometry. For a routine whole body count the whole body geometry calibration is used due to this geometry being the most conservative. A blank resin filled BOMAB is counted periodically and used for background correction. Typical MDA values are 5 nCi for Co-60 and 6 nCi for Cs-137.
9. All analyses are performed in house
10. The efficiency calibration is performed using a solid mixed gamma standard (88 keV to 1836 keV with an optional 59 keV line) contained in a wheaton plastic scintillation vial. The Canberra RMC-II phantom is used and has four optional geometries. The vial can be inserted into any of the four compartments located on the phantom to simulate the following geometries: (1) Whole Body, (2) Lung, (3) Thyroid, and (4) GI tract. The energy calibration method is documented in procedure RPSD-20-02, "Energy/FWHM Calibration of the Whole Body Counter." The efficiency calibration method is documented in procedure RPSD-20-03, "Efficiency Calibration of the Whole Body Counter." The subject stands vertically within the shadow shield while a 600-second scan is taken. The collected spectrum is analyzed by performing a peak search, peak area calculation, background correction, computing the counting efficiency for each found peak, radionuclide identification and computing MDAs for each isotope, and generating a report. A routine Whole body measurement is documented in procedure RPSD-10-02, "Whole Body Measurement."

### Radionuclides Category VII:

8. A portable HPGe detector with approximately a 30% relative efficiency is used for counting radionuclides in the thyroid category. This detector is shielded with lead and may be n-type/or p-type. The detector is placed over the subject's thyroid and counted for 60 seconds. The signals from the detector are sent to an inspector unit, which houses the NIM electronics. The Canberra GENIE software is employed to collect the spectrum and analyze it. The system is energy calibrated using a mixed gamma source. A 5ml glass ampoule and/or a 30ml plastic vial source is used in conjunction with the ANSI N13.30 cylindrical thyroid phantom to generate an efficiency versus energy curve. The analysis consists of a peak search, peak area calculation, background correction, counting efficiency computation for each peak, radionuclide identification and interference correction, MDA computation for each radionuclide, and report generation. A typical MDA for I-131 is 2.5 nCi.
9. All analyses are performed in house
10. The system is energy calibrated using a mixed gamma source. A 5ml glass ampoule and/or a 30ml plastic vial source is used in conjunction with an ANSI N13.30 cylindrical thyroid phantom to generate an efficiency versus energy curve. The observed net counts for each peak are used to compute its counting efficiency. The energy and efficiency pairs are combined to generate an efficiency versus energy curve. The calibration method is documented in procedure RPSD-21-02 "Calibration of the Gamma Spectrometer." The detector is placed on the subject's thyroid and a spectrum is collected for 60 seconds. The collected spectrum is analyzed by performing a peak search, peak area calculation, background correction, counting efficiency computation for each peak, radionuclide identification and interference correction, MDA computation for each radionuclide, and report generation. The measurement procedure is documented per procedure, RPSD-10-03 "Thyroid Measurement."

## EXAMPLE OF A POORLY COMPLETED DIRECT APPLICATION FOR RADIOBIOASSAY

### Direct Radiobioassay Application

For application form item 7, the following clarifications are included to avoid confusion concerning the desired testing categories and radionuclides:

Transuranium elements via L x-ray in Lungs (Plutonium-238)

Americium-241 in Lungs

**Thorium-234 in Lungs**

Uranium-235 in Lungs

Fission and activation products (Cesium-134 and 137) in Total Body

Both Applications

For information required by application forms items 8 and 10, a copy of the revised Internal Dosimetry Technical Basis Manual is enclosed. The tables below provide quick reference to individual topics.

For application forms item 9, all indirect and direct radiobioassay services subject to DOELAP Accreditation are provided in-house at [REDACTED]

For application forms item 11, a copy of the Internal Dosimetry QA Plan is enclosed.

Indirect (*In Vitro*) Radiobioassay

		Technical Basis Manual (chapter-page)
<u>Item 8</u>	Summary of Important Features	10-1,2
	Type of Counting System	10-9,10,15,16
	Counting Configuration	10-10,11,15,16
	Data Reduction	10-11 to 17
	MDA	5-15; 10-13 to 17
	Peak Identification	10-11 to 14,17
	Energy Calibration	10-10,11
<u>Item 10</u>	Efficiency Calibration	10-10,11
	Routine Counting Procedures	10-14,15

Direct (In Vivo) Radiobioassay

		Technical Basis Manual (chapter-page)	
		<u>Whole Body</u> <u>Counting</u>	<u>Chest</u> <u>Counting</u>
<u>Item 8</u>	Detector Type	11-1	11-9
	MDA	5-15;11-8	5-15;11-16
	Counting Configuration	11- 4,5,6	11- 9 to 11
	Shielding	11-5	11-2,3
	Data Reduction Algorithm	11-8,16 to 18	11-16,17,18
	Energy Calibration	11-6,7	11-13,14
	Peak Identification	11-8,18,19	11-18,19
<u>Item 10</u>	Efficiency Calibration	11-6,7	11-13,14,15
	Routine Counting Procedures	11-4	11-10,11
	Protocol Differences	-	-



7. *For each system listed in 6., describe important design features, including: detector type, counting configuration, shielding, and data reduction algorithms.*

The Carlebad Environmental Monitoring & Research Center (CEMRC) provides direct radiobioassay service to the [REDACTED] project. Lung and whole body measurements at the CEMRC are simultaneously performed with counting subject positioned horizontally using two arrays of hyper-pure germanium (HPGe) detectors and a specially designed counting bed and positioning mechanism. This bed was designed to allow for simultaneous lung and whole body counts. The bed is constructed from low background steel and provides minimal attenuation of photons between the counting subject and whole body counting detectors (8% attenuation at 662 keV). The positioning mechanism for the lung counting detectors was designed to support four, 3800 mm<sup>2</sup> detectors mounted in two cryostats. The mechanism provides for longitudinal and vertical positioning as well as independent lateral angle and longitudinal tilt adjustment between the two cryostats. Sufficient positioning flexibility is provided such that other body loci, such as skull, liver, wounds, and thyroid, can be examined.

The CEMRC's *in vivo* facility employs two types of HPGe detectors, low energy (LEGe) and coaxial (COAX). The primary function of the LEGe detectors is lung counting; however, the high energy performance (2.1 keV FWHM resolution at 1330 keV) was matched to that of the COAX detectors to allow for both low and high energy counting. Each LEGe detector has an ultra-thin boron-implanted P outer contact on the front face and cylindrical wall. Lithium diffused on the rear surface is the N contact. The detector is fitted with a 0.6 mm thick carbon composite entrance window which provides the maximum mechanical protection with minimal attenuation, without the contamination from primordial radionuclides that is typically present in other window materials (e.g. beryllium). The active diameter, area, and thickness of the LEGe detectors are 70 mm, 3800 mm<sup>2</sup>, and 20 mm, respectively. The four LEGe detectors are arranged in groups of two for use on each side of the chest. The two detectors of each group are incorporated into a single, multi-attitude, 7-liter chryostat.

The second type of detectors employed by the CEMRC (COAX detectors) are used to measure high energy photons (energies > 200 keV) emitted from radionuclides deposited in the whole body. The detectors are essentially cylinders of HPGe with an N contact on the outer surface, and a P contact on the surface of an interior axial well. The N and P contacts consist of diffused lithium and implanted boron, respectively. The inner contact is about 0.3 μm thick. The HPGe has a net impurity level of 10<sup>10</sup> atoms cm<sup>-3</sup>. This trace level of impurities results in depletion of the entire detector volume with moderate bias, resulting in maximum efficiency of charge collection. The active diameter, length, and relative efficiency of the COAX detectors are 75 mm, 76 mm, and 80%, respectively. Each of the four COAX detectors are independently coupled to a 15-liter chryostat. The COAX detectors are positioned in a close, four detector array. This array is positioned behind the lower thorax of the counting subject.

The counting shield consists of large shielded room measuring 9 feet wide, 10 feet long, and 9 feet high. It is constructed from 10-inch thick cast iron (Fe) obtained from pre-World War II iron. The door to the counting shield weighs in excess of 6 tons. It is operated using an air-over-oil hydraulic system. Open, close, and stop controls are provided inside and outside the shield. The hydraulics are configured such that the door defaults to the open in the case of a power failure. A 60 gallon air tank is maintained at 120 pounds per square inch (PSI) to provide reserve pressure to open the door following the loss of power. Conditioned air is provided to the shield from the CEMRC's centralized heating and cooling units. The air is HEPA filtered just prior to entering the room to remove any particulate that may contain radioactive contamination. In addition, an oxygen monitor

**ATTACHMENT 1**  
**INFORMATION FOR ITEMS 7 - 9 OF DIRECT RADIOBIOASSAY DOELAP APPLICATION**

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is periodically used to monitor oxygen levels in the room. A graded-Z liner (Z represents the charge of an element) consisting of 0.25-inch lead, 0.125-inch tin, and 0.125-inch stainless steel was added to the inside of the thick Fe walls of the shield, to attenuate the photons that are produced through cosmic ray interactions within the shield walls.

The software package ABACOS Plus from Canberra industries is used for routine calibration, operation, and data analysis and archival. This software was specially developed for *in vivo* applications and is currently employed at *in vivo* facilities located at General Electric Nuclear, Wilmington, North Carolina; Savannah River Plant, Aiken, South Carolina; Rocky Flats Environmental Technology Site, Golden, Colorado; Oak Ridge National Laboratory, Oak Ridge, Tennessee; Mound Site, Mound, Ohio; Fernald Site, Fernald, Ohio; and Lawrence Livermore National Laboratory, Livermore, California. The ABACOS software allows the choice of several different spectral analysis programs including standard, library-driven, singlet, region-of-interest and Gamma-M peak searches. CEMRC is currently using the standard peak search algorithm. This analysis program was selected because the algorithm is industry-proven and straight forward. Peak determination is accomplished through use of a correlation function that is used to calculate a correlation value for each channel in the spectrum as it is scanned. A correlation value in the discrete case of a multi-channel spectrum is very similar to the second derivative in the continuous case. Therefore, as the algorithm is sampling a portion of the spectrum that contains no peaks, the correlation value should be very close to zero. When a peak is encountered, the correlation value will become positive and remain so until an inflection point is reached. At an inflection point, the correlation value will become negative and increase to a very large, negative value very near the apex or centroid of the peak. When these conditions occur within the spectrum, a peak is considered located. Once a peak is located, the background is subtracted and the peak's net area is calculated using the following Gaussian equation.

$$Area = h \times s \times \sqrt{2\pi} \quad (\text{equation 1})$$

where

$h$  = height of peak

$s$  = width of the peak (keV)

The Gaussian equation is fitted using a weighted least-squares method where the weighting factor is the inverse of the variance in the photopeak data. The net area is then compared to the decision level, which tests the statistical significance of the peak when compared to the expected variance in background. Net peak areas greater than decision level are considered statistically significant at  $\alpha = 0.05$ . Decision level (DL) is calculated using the following equation as recommended by ANSI 13.30, Performance Criteria for Radiobioassay.

$$DL = 2.33\sigma_b \quad (\text{equation 2})$$

where

$\sigma_b$  = standard deviation of background

8. ***For each category state whether it is processed in-house, in a commercial laboratory, or in another government facility or laboratory.***

**ATTACHMENT 1**  
**INFORMATION FOR ITEMS 7 - 9 OF DIRECT RADIOBIOASSAY DOELAP APPLICATION**

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All direct radiobioassay measurement categories are performed by a commercial laboratory, the Carlsbad Environmental Monitoring & Research Center.

**9. Describe the calibration and routine counting procedures used in the direct radiobioassay measurement. Indicate protocols that may differ for different geometries.**

The first successful calibration phantom for the actinide radioisotopes was the Humanoid torso developed at Lawrence Livermore National Laboratory (LLNL) in 1981. The Humanoid phantom is a simulation of a human torso and is used to calibrate the lung counting arrangements. The original phantom was constructed by LLNL to be identical to a specific cadaver. LLNL constructed only a small group of duplicates, so later torso phantoms were constructed by Radiology Support Devices (RSD). CEMRC's phantom was purchased from RSD. The phantom is constructed of tissue equivalent plastic with a simulated skeleton for anatomical and radiological correctness, including both scattering and shielding. The phantom has lungs, heart, liver, stomach, and tracheobronchial lymph nodes that are anatomically and radiologically correct. The inert organs are replaced with active organs containing known amounts of radioactive material for calibration purposes. The lungs of the Humanoid phantom are similar in size and density to human lungs. CEMRC has 8 individual lung sets containing a known amount of the radioisotopes Pu-238, Pu-239, Am-241, natural uranium, enriched uranium, and Am-241/Eu-152. Each lung set has a detailed certificate and is traceable to the National Institute of Standards (NIST).

The low energy photons emitted by the actinides are significantly attenuated by chestwall tissue. In order to calibrate for variations in chestwall tissue thickness, the Humanoid phantom has three sets of four chest plates, each differing in chestwall composition. The chest plates are used singly and not in combination. Three sets of the chest plates are available to simulate: 1) 87% adipose and 13% muscle, 2) 50% adipose and 50% muscle, and 3) 100% ICRP muscle. Use of the bare phantom and the chest plates give chestwall thickness calibration ranges of 1.7 cm to 4.2 cm.

Bottle manikin absorption phantom (BOMAB) is the "standard" calibration phantom for fission products distributed in the whole body and is used to calibrate the whole body arrangements. The BOMAB consists of a group of polyethylene bottles that are arranged to simulate the body of reference man. There are 10 bottles that represent the head, neck, upper body, pelvis, two thighs, two lower legs, and two arms. The phantom contains about 58 liters of liquid. The phantom is made radioactive by adding a uniform concentration of a NIST traceable mixed gamma source containing Cd-109, Co-57, Ce-139, Hg-203, Sn-113, Y-88, Cs-137, and Co-60 to each of the bottles.

The energy alignments for the low energy and high energy signal processing chains are 8 to 250 keV in 4096 data channels (approximately 0.061 keV/channel) and 100 to 2000 keV in 4096 channels (approximately 0.49 keV/channel), providing approximately 90 and 20 channels per photopeak area, respectively. This energy alignment provides an adequate number of channels within a photopeak for Gaussian fitting routines where a minimum of 7 channels are required. Energy alignment is performed at the time of calibration, and may include ADC zero adjustments and amplifier gain adjustments. During routine operations only the amplifier gain is normally adjusted if energy realignment is necessary as indicated by quality control data.

**ATTACHMENT 1**  
**INFORMATION FOR ITEMS 7 - 9 OF DIRECT RADIOBIOASSAY DOELAP APPLICATION**

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The calibration procedure establishes predictive equations for MCA channel location and resolution as a function of photon energy for individual detectors as well as summed arrangements, whole body counting arrangement efficiency as a function of photon energy, and lung counting arrangement efficiency as a function of photon energy, chestwall thickness, and chestwall composition. The first step in the procedure is to calibrate for individual detector energy and resolution. Following energy alignment a calibration spectrum is acquired for each detector using standards that produce multi-photopeaks distributed throughout the analysis energy range. Individual peak location (centroid) and resolution (FWHM) are determined using equation 2 for each photopeak in the calibration spectra. Peak location calibration (commonly referred to as energy calibration) is accomplished by regressing individual peak centroids against MCA channel using the following equation:

$$E = A_1 + A_2C + A_3C^2 + A_4C^3 \quad (\text{equation 3})$$

where:

$E$  = photon energy (keV)

$A_i$  = regression coefficients

$C$  = photopeak centroid channel

Resolution calibration is accomplished similarly to that of energy using the following equation:

$$F = B_1 + B_2\sqrt{E} \quad (\text{equation 4})$$

where:

$F$  = FWHM (keV)

$B_i$  = regression coefficients

$E$  = photon energy (keV)

Once the individual detectors are calibrated for energy and resolution, this process is then repeated for the summed arrangements.

The final step in the process is efficiency calibrations for lung and whole body counting arrangements. Calibrations are performed on the summed spectra of the individual detectors in each arrangement. A calibration spectra is acquired for each arrangement using the previously described calibration phantoms. Net peak areas are determined for each photopeak using equation 1.

For the whole body counting arrangement, efficiency is determined by regressing summed response (ratio of net peak area and total photon emissions) against photon energy using the following equation:

$$\varepsilon = e^{a_2 + a_3x + a_4x^2 + a_5x^3 + a_6x^4 + a_7x^5} \quad (\text{equation 5})$$

**ATTACHMENT 1**  
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where:

$\varepsilon$  = efficiency (counts  $s^{-1}$  per gamma emission  $s^{-1}$ )

$a_i$  = regression coefficients

$x$  = average calibration energy (keV)

The average calibration energy is the sum of the lowest and highest photon energies used for calibration divided by two.

For the lung counting arrangements, a family of equations based on equation 5, is created for each chestwall thickness for a given overlay set composition (e.g. 100% ICRP muscle). The following equation is then used to interpolate efficiencies for chestwall thickness between phantom calibration points:

$$\varepsilon = a_1 e^{(-a_2 CWT)} \quad (\text{equation 6})$$

where:

$CWT$  = chestwall thickness (cm)

$a_i$  = regression coefficients

The software does not currently interpolate between chestwall composition type. If this calculation is necessary, it will be performed using an equation similar to equation 6.

Routine counting consists of counting subjects reporting to the *in vivo* monitoring facility at their appointed time. They are directed to change out of their street clothes and into a cloth garment provided by the CEMRC. If the individuals have entered a radiological boundary area, they are responsible for showering prior to arrival at the CEMRC. If the subject is excessively dirty, they may be asked to shower prior to their count. These precautions are necessary to prevent contamination of the room with naturally occurring radioactive materials. The subject's height and weight are measured and recorded for input into the chestwall thickness algorithm. In addition, the subject chestwall may be measured using ultrasonic techniques. The subject is positioned on the bed so that the COAX detectors are positioned behind the lower thorax and the lung counting detectors are placed over the chest. The lung counting detectors are adjusted with the face of the detectors parallel with, and as close to as practical, the surface of the chestwall. The upper edges of the detectors are just below the lower edges of the collar bones. Routine count time for both lung and whole body measurements are 1800 seconds.

Quality control measurements are performed twice daily in accordance with ANSI 13.30. Quality control charts are plotted in order to verify that the system is routinely performing as it was when calibrated. Quality control charts are created to track individual detector performance, including peak centroid and resolution at two extremes in analysis range, background, and net peak area for a standard source. Quality control charts are also created for summed arrangements including activity calculation of a standard source and background.

### Minimum Detectable Amount

Radionuclide	Organ	<sup>a</sup> MDA (nCi)
<sup>241</sup> Am	Lung	0.24
<sup>235</sup> U	Lung	0.11
<sup>137</sup> Cs	Whole Body	0.48
<sup>134</sup> Cs	Whole Body	0.76

<sup>a</sup>MDA for a routine, 30 minute count.

## WELL COMPLETED PARTS OF AN INDIRECT APPLICATION (demonstrates knowledge about the process!)

**System ID:** Perkin-Elmer DRCE ICP-MS

**System:** The Perkin-Elmer DRCE consists of an Inductively Coupled Plasma-Mass Spectrometer (ICPMS), AS-93 Plus auto-sampler, and data analysis workstation running the Perkin-Elmer ELAN software. The ELAN software is used for all aspects of sample analysis including: control of the auto sampler; calibration; data analysis; and quality assurance. Verification and Validation (V&V) for this software is performed by Perkin-Elmer and documentation of the V&V for the software is maintained near the system. The Perkin-Elmer DRCE includes a Dynamic Reaction Cell which is currently not in use for the uranium analysis. The data analysis workstation is also used as a network interface between the ICP-MS, a network server which stores the data from the ICP-MS, and the Bioassay Laboratory Information Management System (BLIMS). For routine U-238 analyses, after data analysis, analytical results are electronically transferred to BLIMS. For U-235/U-238 analyses, analytical results are manually entered into an Excel spreadsheet where the isotope ratios and relevant combined standard uncertainties (CSU) are calculated. The results from the Excel spreadsheet are manually entered into the BLIMS database.

**Counting Configuration:** After sample preparation, samples are in 15 mL conical tubes. These conical tubes are placed in the auto-sampler and sample is aspirated from each tube for analysis.

**Shielding:** Not applicable.

**Data Reductions Algorithms:** Concentration calculations are performed by the Perkin-Elmer ELAN software. A linear through zero calibration curve is constructed using the response of eight U-238 calibration standards and one blank for routine U-238 analyses and five calibration standards and one blank for U-235/U-238 analyses. The sample concentration determined from these calibration curves is corrected for salt and viscosity variations between the sample and the calibration standards using the U-233 internal standard response. The ELAN software assumes that all isotopes of uranium are affected in the same way as the internal standard.

**Peak Identification:** The ICP-MS operates in a peak-hopping mode and analyzes the peak area for preselected masses.

**Energy Calibration:** The ICP-MS is 'tuned' for optimum response using a U-238 standard.

**Other:** For routine U-238 analyses, a one mL aliquot of the urine sample is pipetted into a 15 mL polyethylene conical tube. 100 uL of U-233 tracer (10ug/L) and 0.5 mL of ultrapure nitric acid are added. The contents are digested in a microwave oven. This treatment converts the uranium, both analyte and tracer, into the uranyl form (UO<sub>2</sub>)<sup>2+</sup>, and ensures complete mixing of the tracer and analyte. The digested samples are diluted with deionized water to 10 mL mixed and analyzed for U-238 using an ICP-MS. For U-235/U-238 analyses a 30 mL aliquot is transferred to a 50 mL polyethylene conical tube. 100 uL of U-233 tracer (10 ug/L) and 5.5 mL

of ultrapure nitric acid are added. The contents are digested in a microwave oven. This treatment converts the uranium, both analyte and tracer, into the uranyl form  $(\text{UO}_2)_2^{2+}$ , and ensures complete mixing of the tracer and analyte. The urine salts are separated using a 2 mL TRU resin cartridge. The uranium isotopes are eluted from the column with 10 mL of ammonium bi-oxalate and analyzed for U-235 and U-238 using an ICP-MS. MDA95s for the U-235/U-238 procedure are: U-235: 0.0000092 ug/L U-238: 0.00054 ug/L

# POORLY COMPLETED PARTS OF AN INDIRECT APPLICATION

**System:** Packard Liquid Scintillation Counters

**Counting Configuration:** Three milliliters of urine and 19 mL Ultima Gold scintillation cocktail in a 25 mL plastic scintillation vial.

**Shielding:** Not applicable

**Data Reductions Algorithms:** Window areas are determined by the Packard software purchased with the instrument. Activity calculations are determined using a program developed in-house.

**Peak Identification:** Not applicable

**Energy Calibration:** Calibration is performed during the system normalization and calibration procedure. This procedure is done approximately weekly by the analyst performing the routine measurements and at least yearly by a Packard service representative

**Other:**

Strontium-89/90 or Strontium-90 0.3 pCi/L

**System:** Tenelec model LB4000 alpha/beta counting system alpha/beta counting system consisting of 16 low background gas flow proportional counters.

**Counting Configuration:** Stainless steel planchet (50 mm diameter, 6 mm deep, in the shape of an inverted truncated cone with a 12 mm diameter flat bottom).

**Shielding:** Commercially available shielding

**Data Reductions Algorithms:** Beta counts are determined by the Tenelec software supplied with the instrument. Calculation of activity is done using a program developed in-house

**Peak Identification:** Not applicable

**Energy Calibration:** Calibration is not applicable because the proportional counter is not operated as an energy spectrometer.