U. S. BIOASSAY INTERCOMPARISON STUDIES PROGRAM AT OAK RIDGE NATIONAL LAB

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Introduction:

The Intercomparison Studies Program (ISP) for in-vitro bioassay located at Oak Ridge National Laboratory (ORNL) has been in place since May 1991. The program was originally created to fill a need (identified in 1990 by a Technical Safety Appraisal) in the Radiobioassay area at ORNL, specifically in the areas of Quality Control, Quality Assurance, and Performance Testing. In the beginning, this consisted of two or three laboratories working in a pilot intercomparison program. Once it was determined that this could work effectively, the program began to seek additional members to broaden the scope of the effort. The program became formalized with a quarterly report in January 1992.

The ISP currently provides cross-check blind/double-blind samples spiked with known amounts of radioactivity to various Department of Energy (DOE) facilities, universities, and private industry organizations throughout the United States. These samples can be packaged according to ORNL procedures (ORNL sample bottles, ORNL chain-of-custody forms, tamper seals etc.), for a single blind sample or according to the needs of a particular facility if the double-blind sample mode is to be maintained. In 1998, the customer base was broadened to include European facilities.

In January 1993, the whole-body count program was added. This involves each participating facility receiving a "block" phantom from the ISP and determining a geometry factor using a known standard. (Each facility keeps its own "block" phantom as long as they remain a participant in the program). At quarterly intervals, each participant receives an unknown sample for analysis. The sample is counted and the data is collected for publication in an annual report.

In October 1994, the fecal program was added. This involves spiking an artificial matrix with known amounts of radioactivity. Laboratories receive unknown samples on a quarterly basis. The sample is counted and the data is collected and published in a quarterly report.

The ISP Laboratory is set up to function in the same manner as a routine bioassay facility. This includes having the capability to perform all the separations and analyses that would be carried out in a typical radiobioassay laboratory, along with the necessary policies, procedures, and document controls that go along with these techniques. The ISP also maintains archive samples which can be analyzed in the QC laboratory at the request of any of the participants if a conflict or discrepancy in a sample analysis/result occurs.

Urinalysis Program:

Participation in the (ISP) urinalysis program is usually on a monthly basis. Urine is collected and frozen until it is ready for use. Each month approximately fifty liters is thawed, and pooled in a carboy for sample preparation. The fifty liters is divided into sample batches. (Typically a batch can be anywhere from 2-15L). Each batch is spiked with a known amount of radioactivity from an ISP standard and is allowed to equilibrate overnight. The radionuclides (spikes) used are all National Institute of Standards and Technology (NIST) traceable and the subsequent primary, secondary and tertiary standards prepared are done so using a semi-micro balance which has been calibrated and functional checked using NIST traceable weights. After equilibration, the batch is then divided into one liter samples, tagged by ORNL Health Physics and shipped to the various participants in the program. Below is a list of the radionuclides and the spike levels that are commonly used in the program:

Th-nat:	less than 1 dpm/liter
Th-nat:	1-5 dpm/liter
Th-nat:	5-15 dpm/liter
U-nat:	less than 1 dpm/liter
U-nat:	1-5 dpm/liter
U-nat:	5-15 dpm/liter

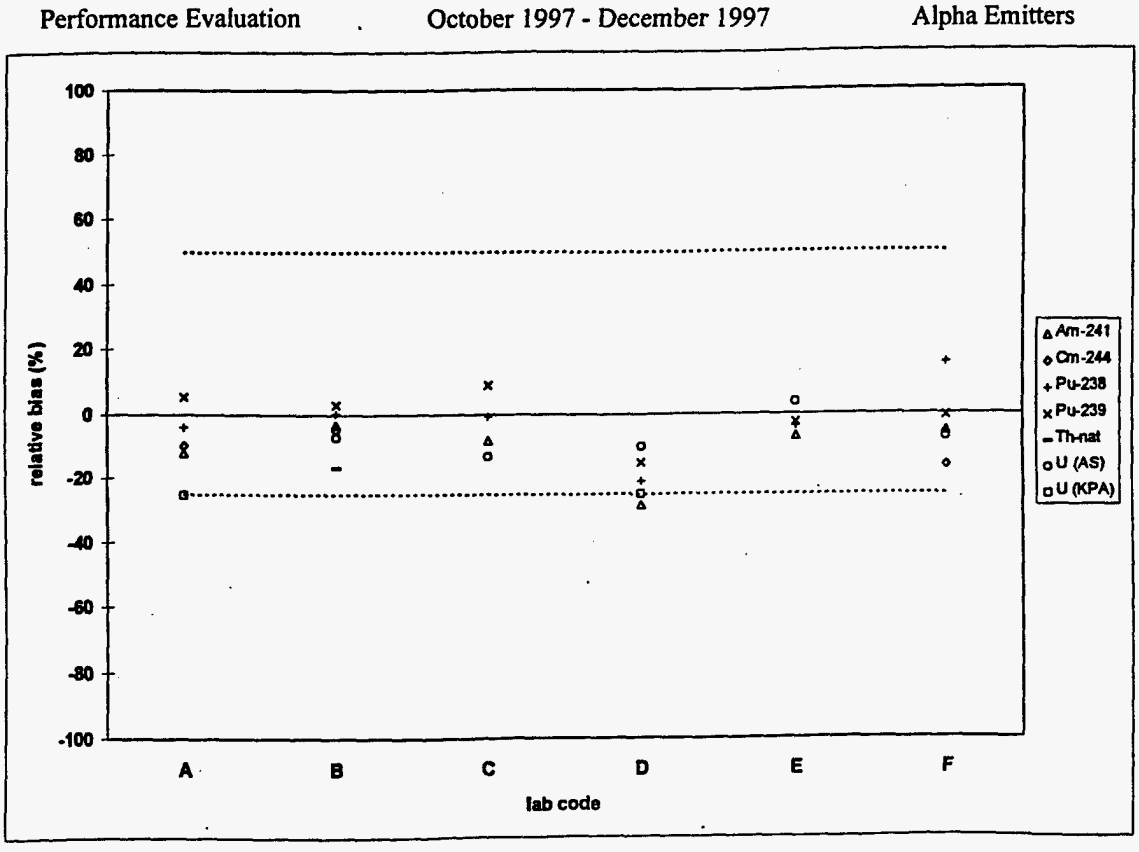
DISCLAIMER

Pu-238:	less than 1 dpm/liter
Pu-238:	1-5 dpm/liter
Pu-239:	less than 1 dpm/liter
Pu-239:	1-5 dpm/liter
Am-241:	less than 1 dpm/liter
Am-241:	1-5 dpm/liter
Cm-244:	less than 1 dpm/liter
Tc-99:	50-2000 dpm/liter
P-32:	50-1000 dpm/liter
Sr-90:	5-100 dpm/liter
Sr-90:	50-500 dpm/liter
H-3:	less than 250 dpm/mL
H-3:	250-500 dpm/mL
C-14:	5-500 dpm/mL
S-35:	5-500 dpm/mL
Co-60:	50-1000 dpm/liter
Cs-137:	50-1000 dpm/liter

All radionuclides are NIST traceable standards prepared by NIST with the following exceptions: Th-Nat is an NIST traceable standard prepared by Analytics, C-14, S-35, and P-32 are NIST traceable standards prepared by Amersham. Blank samples (urine samples which have not been spiked with radioactivity) are also provided as cross-check samples. In addition, samples which are outside the above ranges are provided, but this is usually not included in the formal intercomparison program.

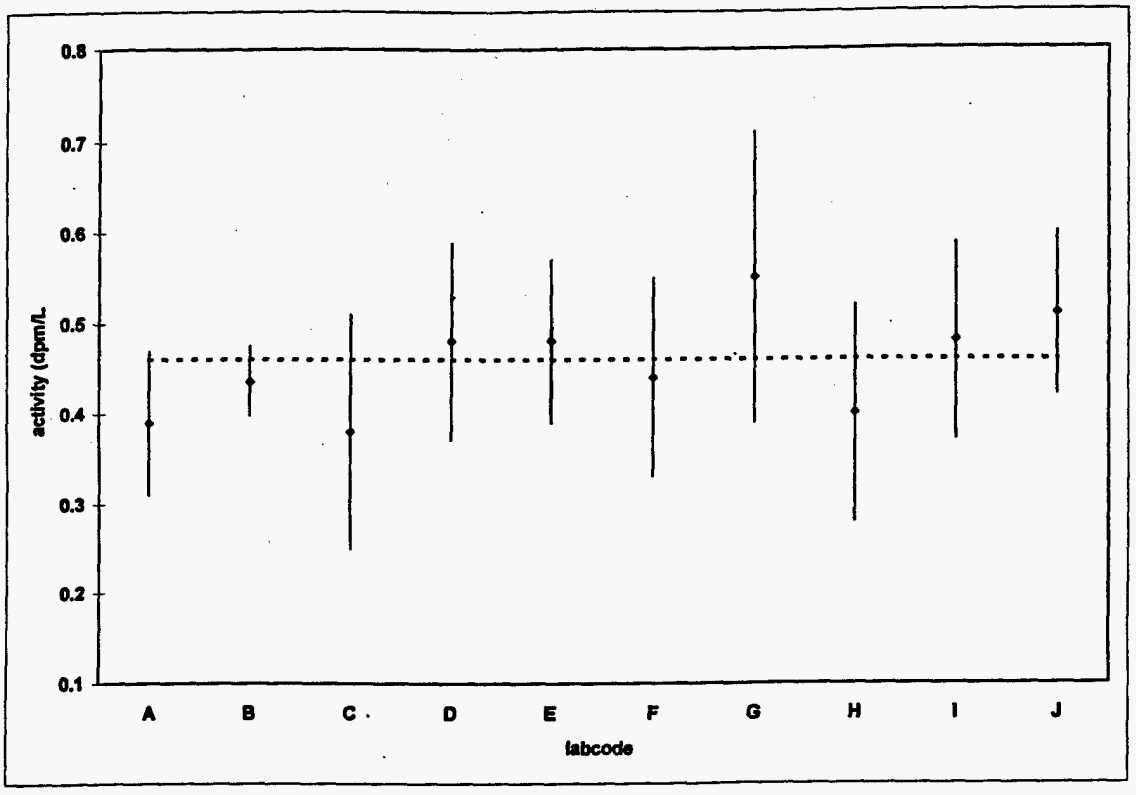
The samples are analyzed by each participant, and the data is sent to ISP for statistical analyses. Below are examples of the types of statistical analyses/results. A report consisting of relative bias data for each radionuclide range that has been tested in the last quarter (Fig. 1), and

FIG. 1 RELATIVE BIAS CHART



an intercomparison of laboratory performance for each individual analysis (Fig. 2). These are published once per quarter.

FIG. 2 HI-LO CHART
Intercomparison Results
Pu-239
January 1997



multiple entries for a single lab indicate a split sample

number of participating	7	result mean	0.45
number of reported results	10	result standard deviation	0.06
incomplete, lost in process, or <MDA	0	result median	0.46
known value	0.46	% deviation mean	-1.15
		normal mean	-0.10
		% deviation median	0.00
		normal median	0.00

Note: The quarterly report is published three months after the last spike sample to be included in the report has been shipped to the service laboratory. This is so that the service laboratories have time to process their samples, complete their analyses, and relay the data to the ISP.

Fecal Program:

Participation in the fecal program is usually on a quarterly basis. The samples are prepared using an artificial matrix[1] (see Table 1.) The mass of a sample is typically 30g. The preparation of the fecal samples (batches) is a three step process: The dry chemicals are mixed together in a large container and retained. The amount of water needed per batch is calculated and this water is spiked with a known amount of radioactivity from an ISP standard(s). A portion of the spiked water (10g) and a portion of the dry mixture (19g) are then combined in a plastic bag, along with a measured amount of oleic acid (1g) to make up a sample. This process is repeated for each sample in the batch. The plastic bag is heat sealed and placed inside a second bag which is also heat sealed. The fecal samples are tagged by ORNL Health Physics for shipping to the various participants in the program.

TABLE 1.

CHEMICAL COMPOSITION OF SYNTHETIC FECAL SAMPLE

Chemical Component	Approximate g/sample
Calcium Carbonate	**0.64**
Ferric Ammonium Sulfate	**0.03**
Magnesium Carbonate	**0.40**
Potassium Carbonate	**0.55**
Ammonium Dihydrogen Phosphate	**1.33**
Sodium Sulfate	**0.24**
Ammonium Chloride	**0.03**
Zinc Sulfide	**0.01**
Stannous Chloride	**0.02**
Leucine	**4.64**
Lysine	**3.31**
Methionine	**0.53**
Threonine	**1.32**
Palmitic Acid	**1.98**
Stearic Acid	**1.32**
Oleic Acid	**1.00**
Cellulose	**2.65**
Water containing spike	**10.00**

The radionuclides (spikes) used are all NIST traceable standards prepared by NIST and the subsequent primary, secondary and tertiary standards prepared are done so using a semi-micro balance which has been calibrated and functional checked using NIST traceable weights. Below is a list of the radionuclides and the spike levels that are commonly used in the program:

U-nat:	5-15 dpm/sample
U-nat:	1-5 dpm/sample
Pu-238:	<1 dpm/sample
Pu-238:	1-5 dpm/sample
Pu-239:	<1 dpm/sample
Pu-239:	1-5 dpm/sample
Am-241:	< 1 dpm/sample
Am-241:	1-5 dpm/sample
Th-Nat:	1-5 dpm/sample
Th-Nat:	5-15 dpm/sample

Blank samples (fecal samples which have not been spiked with radioactivity) are also provided as cross-check samples. In addition, samples outside the above ranges may be provided, but this is usually not included in the formal intercomparison program.

The samples are analyzed by each participant, and the data is sent to ISP for statistical analyses. The format that is used for statistical analysis in the fecal program is identical to that used in the urine program (See Figs. 1 and 2).

Whole Body Count Program:

The ISP whole body count program began January 1993. The program provides Co-57, Ba-133, Y-88, Co-60, and Cs-137 cross-check samples in a 0.1M hydrochloric acid matrix (five quart bottles/sample set) which are inserted into identical anthropomorphic phantoms. A known amount of activity is initially provided for each radionuclide to calibrate the detector for this phantom geometry. The preparation of the whole body count samples is a two step process: The amount of water needed per sample set is calculated and this water is spiked with a known amount of radioactivity from an ISP standard(s). (Each tracer addition is made by mass and the current testing range is 10-500 nCi). A portion of the spiked water (10g) is then added to each of the five quart bottles and each bottle is filled with 0.1M hydrochloric acid. This process is repeated for each participant. Each of the bottles is placed inside a plastic bag which is sealed and the whole is placed inside a second bag which is also sealed. The whole body count samples are tagged by ORNL Health Physics for shipping to the various participants in the program. Each participant receives its own individual phantom, which they keep as long as they continue to participate in the program. Cross-check samples are sent to each facility every three months and an annual report is published at the end of the year. Additional radionuclides can be added at the request of any of the participating facilities.

All radionuclides are NIST traceable standards. Co-60 and Cs-137 are prepared by NIST; Co-57, Ba-133, and Y-88 are prepared by Amersham.

The format that is used for statistical analysis in the whole-body count program is identical to that used in the urine program (See Figs. 1 and 2).

Sample Load:

The expected sample load for 1998 for ISP is as follows:

Urinalysis Program:	850-900 samples.
Fecal Program	150-200 samples
Whole body count Program	30-40 sample sets

Advantages to participation in ORNL bioassay intercomparison programs:

- Participation in an established intercomparison (QC) program which has positive recognition from the United States DOE.
- Performance criteria as established by applicable Code of Federal Regulations (CFRs), other standards, and DOE Orders.
- Positive recognition by internal and external auditors.
- Assurance to workers and public that dose exposure readings are accurate and reproducible.
- QA documentation, Performance Evaluation Reports, fully-controlled ISP Procedures, and NIST certificates to attest to performance of laboratory.
- A credible and defensible external QC program in the event of litigation.
- Intercomparison of analysis capabilities between all major DOE contractors.
- Long and short-term trending of analysis performance.
- Current participation by ISP in DOE-sponsored Quality Assurance Program (QAP) and Environmental Protection Agency (EPA) performance evaluation programs to confirm accuracy of internal measurements.

Future Plans:

- ISP is working to become a "Reference Laboratory for Bioassay" per ANSI N42.23[2], "Measurement and Associated Instrumentation Quality Assurance for Radiobioassay Laboratories."
- ISP is hoping to expand into international markets.

References

1 MacLelland, J. A., R. J. Traub, and D. R. Fisher. 1988. Performance Testing of Radiobioassay Laboratories: In Vitro Measurements, Final Report. PNL-6490, Pacific Northwest Laboratory, Richland, Washington.

2 American National Standards Institute (ANSI). 1996. Measurement and Associated Instrumentation quality Assurance for Radiobioassay Laboratories. ANSI N42.23-1996, New York.

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