U.S. DEPARTMENT OF ENERGY

PNNL-21518

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

RESULTS OF THE EXCRETA BIOASSAY QUALITY CONTROL PROGRAM FOR APRIL 1, 2009 THROUGH MARCH 31, 2010

CL Antonio

July 2012

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Pacific Northwest National Laboratory
Richland, Washington 99352

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Cheryl L. Antonio

February 2010

Peer Reviewed by _____ 6/28/12
Jay Maclellan Date

SUMMARY

A total of 58 urine samples and 10 fecal samples were submitted during the report period (April 1, 2009 through March 31, 2010) to General Engineering Laboratories, South Carolina by the Hanford Internal Dosimetry Program (IDP) to check the accuracy, precision, and detection levels of their analyses. Urine analyses for Sr, ^{238}Pu, ^{239}Pu, ^{241}Am, ^{243}Am, ^{235}U, ^{238}U, elemental uranium and fecal analyses for ^{241}Am, ^{238}Pu and ^{239}Pu were tested this year as well as four tissue samples for ^{238}Pu, ^{239}Pu, ^{241}Am and ^{241}Pu. The number of QC urine samples submitted during the report period represented 1.3% of the total samples submitted. In addition to the samples provided by IDP, GEL was also required to conduct their own QC program, and submit the results of analyses to IDP. About 33% of the analyses processed by GEL during the third year of this contract were quality control samples. GEL tested the performance of 21 radioisotopes, all of which met or exceeded the specifications in the Statement of Work within statistical uncertainty (Table 4).

IDP concluded that GEL was performing well for all analyses tested, and concerns identified earlier were satisfactorily resolved (see section on Follow-up on Concerns During the Fourth Contract Year).

Beginning in May 2006, it was decided to evaluate the MDA capability of the Lab based on detections of samples spiked at the CL level rather than on blanks, with the exception of ^{238}Pu and ^{243}Am. The decision not to submit blank samples, other than for ^{238}Pu and ^{243}Am, was made in order to increase the number of samples spiked at the CL and therefore improve the statistics for evaluating MDA, bias and precision. The MDA criteria would be met if less than 20 percent of the reported results for samples spiked at the Contractual Detection Level are less than the decision level (for n between 5 and 25) or less than 10 percent of the reported results are less than the decision level (for $n > 25$).

The isotopic uranium analysis reports on three uranium isotopes: ^{234}U, ^{235}U, and ^{238}U. The isotopes are differentiated only during counting by alpha spectrometry. All performance criteria were met, the relative bias reported by GEL was within statistical uncertainty and determined to be acceptable.

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, ^{233,234}U was not evaluated. IDP submitted 10 urinalyses samples throughout the year for isotopic uranium analysis. The performance statistics for ^{235}U and ^{238}U were reviewed and the MDA for ^{235}U and the bias and precision for ^{238}U were acceptable.

No concerns were identified with the uranium mass (^{238}U-ICPMS) urinalysis program and it was considered acceptable. Because IDP uses a 0.2 µg screening level for elemental uranium, samples spiked at 0.06 µg were discontinued. The MDA at the contractual level of 0.06 µg was evaluated through GEL's program and was found to be acceptable. The relative bias and precision were likewise acceptable. The bias and precision from the 17 samples submitted IDP met the acceptance criteria. The bias and precision was tested by IDP at 0.2 µg and by GEL at 1 µg/L and at 0.05/L µg.

In February 2009, the KPA uranium mass analysis was being phased out and replaced with the ICPMS analysis for ^{238}U, which comprises 99% of the uranium isotopic mixture by mass.

During the first quarter GEL was processing both elemental uranium via KPA and ICPMS for ^{238}U. Sample results reported in the first quarter for ^{238}U via ICPMS initially were reported without a corresponding analyzed volume. Because GEL reported the analyzed volume in liters and the database rounds to 2 significant figures, the resulting 0.001 L analyzed volume was rounded to 0.00 L. GEL changed their programming to reflect milliliters rather than liters. Also, effective June 1, 2009, GEL changed the analyzed volume from 1 ml to 2 ml in order to more comfortably comply with the CL of 0.06 µg/sample.

The total strontium procedure is used to screen samples to determine which will require analysis for ^{90}Sr. Samples with total strontium results less than 15 dpm do not undergo further analysis. Samples with results greater than or equal to 15 dpm may undergo ^{90}Y in growth to specifically determine ^{90}Sr levels. The calculated MDA, reported by GEL and tested by IDP, for the total strontium part of the analysis was less than 55% of the CL. The relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all within limits. The 23 samples spiked at the contractual level by IDP were all detected. The strontium urinalysis procedure was concluded to be acceptable. There was an issue however, in September 2009, with a batch of samples to be analyzed for strontium that were switched with another batch. This is discussed further in the section on follow-up concerns and documentation of the investigation provided in Attachment 2.

Samples spiked with ^{238}Pu and ^{239}Pu were analyzed using the same procedures and same reagents. The two isotopes are differentiated only at the end of the procedure by alpha spectrometry. Therefore, laboratory performance is expected to be similar for both isotopes using any of the seven procedures that incorporate plutonium analysis (IPU, IPA, IPS, IPSA, IPSR, IUPU, and ITPAC).

The MDAs and performance statistics for ^{239}Pu and ^{238}Pu in urine were acceptable. The 20 samples spiked at the CL and the five spiked at greater than three times the CL for ^{239}Pu were reported and all showed detection greater than the decision level. There were 25 blank samples analyzed for ^{238}Pu activity, none of the 25 samples detected activity in excess of the decision level. Overall the plutonium urinalyses were considered acceptable.

The MDA and performance statistics for ^{239}Pu and ^{238}Pu in feces were acceptable. Approximately 15% of the fecal samples analyzed were duplicated to test the consistency of the aliquoting procedure. A review of the duplicate samples determined that the aliquoting procedure produced results within 3 sigma of the initial results. The fecal aliquoting procedure was acceptable. This year IDP submitted 5 actual fecal samples spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The precision and bias for ^{239}Pu and ^{238}Pu met the performance criteria. The performance statistics reported by GEL for ^{239}Pu met the acceptance criterion. The low-yield rate and failed analysis rate for fecal sampling over the contract year was 6% and 2% respectively, which was below the contractual level of 10%. The problem of technician errors resulting in a failed analysis rate greater than 10% seems to have been corrected. Overall the plutonium fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

The ^{241}Am fecal and urine analysis met the acceptance criteria for MDA, relative bias and precision. The MDA as reported by GEL and tested by IDP was less than 50% of the contractual level. All 18 of the ^{241}Am samples spiked at the contractual detection level (CL) and the 5 spiked at greater than three times the CL were detected. The relative bias and precision as reported by GEL

and tested by IDP met the performance criteria. The current AM241 urinalysis procedure was considered acceptable.

The ^{241}Am fecal duplicate samples were evaluated and it was concluded that the aliquoting procedure produced results within the control limits. This year IDP submitted 5 blank fecal samples and 5 fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. The failed analysis rate for ^{241}Am fecal analyses was 2%, which was below the contractual level of 10%. The problem of technician errors resulting in a failed analysis rate greater than 10% seems to have been corrected. Overall the ^{241}Am fecal analyses were considered acceptable.

The four tissue samples submitted for ^{238}Pu, ^{239}Pu, ^{241}Am and ^{241}Pu analysis, showed results consisted with direct measurements. The analysis of the tissue samples not only tested skin tissue analysis but also the expedite processing, see Attachment A for details.

The AM243 procedure was identical to the AM241 procedure, except a different tracer is used (^{244}Cm instead of ^{243}Am). The seven blank ^{243}Am QC samples submitted were all reported with results less than the decision level and the calculated MDA was 35% of the contractual detection level. The performance statistics for ^{243}Am, as tested by GEL, met the acceptance criteria. The ^{243}Am procedure was concluded to be acceptable.

IDP did not submit QC samples to test the isotopic curium program, therefore performance statistics were based on the GEL QC results. GEL tested the MDA for ^{242}Cm and ^{244}Cm and the relative bias and precision for ^{244}Cm. The results met the acceptance criteria and the isotopic curium urinalysis program was considered acceptable.

IDP also did not submit QC samples to test the isotopic thorium program, therefore performance statistics were based on the GEL QC results. GEL tested the MDA for ^{228}Th, ^{229}Th, ^{230}Th and ^{232}Th and the relative bias and precision for ^{232}Th. The results met the acceptance criteria and the isotopic thorium urinalysis program was considered acceptable.

A new ^{236}U analysis procedure was initiated in June 2007 and the procedure was formally approved in June 2008. The analysis for ^{236}U uses inductively coupled plasma mass spectrometry. IDP submitted ten blank samples and the MDA were found to be acceptable. The MDA and relative bias and precision reported by GEL met the performance criteria. The ^{236}U analysis procedure was considered acceptable.

CONTENTS

TABLES

INTRODUCTION

This report summarizes the results of the excreta bioassay quality control programs monitoring of the performance of General Engineering Laboratories (GEL) for samples submitted from April 1, 2009 through March 31, 2010. During the reporting period GEL analyzed, under contract 11530 effective date ending 2/23/10 and contract 112512 effective date 2/24/10 with Battelle, 4980 urine and 42 fecal samples for various radionuclides. This is about the same workload as reported in the 2008 report.

The results of the analyses are part of a system of legal records concerning internal deposition of radionuclides for workers at the Hanford Site. GEL is required to have a rigorous quality control (QC) program to ensure the accuracy of its results. In addition, the Pacific Northwest National Laboratory's (PNNL) Hanford Internal Dosimetry Program (IDP) has a QC program in place to independently check the accuracy of the results from GEL. The objective of the PNNL excreta bioassay QC program is to provide quantitative data to support the assessment of performance criteria for excreta bioassay analyses, as specified in the Statement of Work (Battelle 2009).

The reliability of the excreta bioassay program depends, to a significant extent, on the adoption and implementation of performance criteria for laboratory accuracy, precision, and detection levels. Such performance criteria are established in the Statement of Work (Battelle 2007) and include the following:

- Actual minimum detectable activities (MDAs) determined from QC samples for the year shall be equal to or less than the contractual detection level (CL) in the Statement of Work, as calculated from blank QC samples.
- The mean relative bias, B_r, shall fall within ± 20% when calculated from 15 to 50 samples spiked at greater than three times the CL, and within ± 10% when calculated from greater than 50 samples.
- The relative precision statistic, S_B, shall be less than or equal to 0.4 for samples spiked at greater than three times the CL, and less than or equal to 0.5 for samples spiked between one and three times the CL.

Formulas for MDA, B_r, and S_B, presented in the next section of this report, are based on recommendations in the Health Physics Society (HPS) Standard N13.30 (1996) and are listed in the Statement of Work. In addition to the Statement of Work (SOW) performance criteria, it is expected that the MDA shall also be such that fewer than 10% of the QC samples spiked at the CL shall be reported with values less than the decision level (i.e., twice the total propagated uncertainty of the result).

METHODS

GENERAL METHODS

Urine collected from PNNL employees who are not occupationally exposed to radioactive material was prepared in the 325 Building as blank and spiked samples by PNNL Radiochemical Processing Group (RPG), according to the directions given by the PNNL Internal Dosimetry Program (IDP), following Procedure PNL-MA-565-800-20, Rev. 2. Most samples were submitted as double-blind samples, with the exception of isotopic uranium urinalyses and the spiked fecal samples. Double blind samples are scheduled with and collected by GEL as if they were personnel samples. The isotopic uranium urinalyses were scheduled as single-blind intercomparisons, which meant that GEL was aware they were intercomparison samples but unaware of the activity. The samples were scheduled as single-blinds because they were spiked with a depleted uranium source. Since depleted uranium exposures at Hanford are rare, the intercomparison samples would stand out and the QC alias names used could become known and compromise the double-blind intercomparison program. The spiked fecal samples were artificial fecal samples consisting of a soil matrix. Blank fecal samples were scheduled as double-blind samples and were actual fecal samples.

GEL analyzed urine samples for tritium, ^{90}Sr, ^{242}Cm, ^{244}Cm, ^{238}Pu, $^{239,240}Pu$, ^{241}Pu ^{241}Am, ^{243}Am, ^{228}Th, ^{229}Th, ^{230}Th, ^{232}Th, ^{236}U, ^{234}U, ^{235}U, ^{238}U and elemental uranium and fecal samples for ^{238}Pu, $^{239,240}Pu$, ^{241}Am, ^{234}U, ^{235}U, ^{238}U . To reduce costs in the intercomparison program, plutonium, americium, and strontium analyses were tested using routine sequential procedures when possible (i.e., where one urine sample is analyzed for several radionuclides). The analysis categories specified in the contract with GEL are shown in Table 1. All urinalysis samples contained approximately 1000 ml of urine, except for the samples analyzed for tritium, which contained approximately 100 ml.

GEL's QC sample total is dependent on the number of analytical batches run during the year, and they were well over the 15% criteria specified in the contract.

Table 1: **Battelle Contract 11530 - Feb-06**

Table B-3

Analytical and Reporting Requirements for Routine Processing of Samples

Analysis (Code)	Constituents Reported	Contractual Detection Level (a) (dpm/sample) Urine	Fecal	Determination Time (business days following sample receipt)	Reporting Time Oral[g]	Electronic[a]	Written[a]	Oral Reporting Level; (dpm/sample) Urine	Fecal
Pu(∝) Isotopic (IPU)	Pu-238, Pu-239, 240	0.02	0.2	20	By close of business on day of determination	Within five business days of determination	Within 10 business days of determination	Eq. 1	Eq. 1
Pu(∝) Isotopic (IPUL)	Pu-238, Pu-239, 240	0.005		30				Eq. 1	
Am-241 (AM241)	Am-241	0.02	0.8	20				Eq. 1	Eq. 1
Am-243 (AM243)	Am-243	0.02	0.8	20				Eq. 1	Eq. 1
Cm(∝) Isotopic (ICM)	Cm-242, Cm-244(b)	0.02		20				Eq. 1	
U(∝) Isotopic (IU)	U-233, 234, U-235, U-238	0.02		20				(f)	
Th(∝) Isotopic (ITH)	Th-228, Th-229, Th-230, Th-232	0.1	1	20				Eq. 1	Eq. 1
Tritium (H3)	H-3	20 dpm/ml		5				10dpm/ml	
Sr-total (SR)	Sr (sum Sr-89 + Sr-90)	10		20				5	
Sr-90 (SR90)[c]	Sr-90	10		30				5	
Gamma Spectroscopy (ISPEC)	K-40, Cs-137 + Others(d)	See Table B-5		20				Eq. 1	
Gamma Spectroscopy (LEPD)	Am-241	5		20				Eq. 1	
U-nat (U)	Elemental U	0.06 µg/sample	0.3 µg/sample	20				0.2	0.2
Sequential Analyses:									
Pu(∝) Iso and Sr-total (IPS)	As for individual analyses	As for individual analyses		25	As for individual analyses				
Pu(∝) Iso, Am-241 (IPA)				25					
Pu(∝) Iso, Am-241, Sr-total (IPSA)				25					
Pu(∝) Iso, U-nat (IUPU)				25					
Actinide(∝) Isotopic (ITPAC)[e]				25					
Pu(∝) Iso and U ISO (IPIU)				25					

(a) Time allowed following determination of results to receipt of results by Battelle.

(b) Report measured activity for Cm-246, and Cm-248 upon request of the Battelle Technical Administrator.

(c) If total Strontium is less than 15 dpm, Y ingrowth is not required.

(d) Report all isotopes present at levels exceeding Equation 5. If ordered by the Battelle Technical Administrator, report results for radionuclides in Table B-5 specified in the processing instruction, regardless of the activity measured.

(e) Pu (∝) Isotopic, Am-241, and Cm (∝) Isotopic.

(f) 0.16 dpm for U-234, 0.15 dpm for U-238, and the greater of 0.007dpm and Equation 5 for U-235.

(g) Oral report required only when analytical results exceed level specified. **Eq. 1 Lc=2(combined standard uncertainty)**

Table 1 cont.

Table B-3: Effecitve 1/7/2009

Analytical and Reporting Requirements for Routine Processing of Samples

Analysis (Code)	Constituents Reported	Contractual Detection Level [a] (dpm/sample) Urine	Fecal	Determination Time (business days following sample receipt)	Reporting Time: Oral[g]	Electronic[a]	Written[a]	Oral Reporting Level; (dpm/sample) Urine	Fecal
Pu(∝) Isotopic (IPU)	Pu-238, Pu-239, 240	0.02	0.2	20	By close of business on day of determination	Within five business days of determination	Within 10 business days of determination	Eq. 1	Eq. 1
Pu(∝) Isotopic (IPUL)	Pu-238, Pu-239, 240	0.005		30				Eq. 1	
Am-241 (AM241)	Am-241	0.02	0.2	20				Eq. 1	Eq. 1
Am-243 (AM243)	Am-243	0.02	0.2	20				Eq. 1	Eq. 1
Cm(∝) Isotopic (ICM)	Cm-242, Cm-244(b)	0.02		20				Eq. 1	
U(∝) Isotopic (IU)	U-233, 234, U-235, U-238	0.02		20				(f)	
Th(∝) Isotopic (ITH)	Th-228, Th-229, Th-230, Th-232	0.1	1	20				Eq. 1	Eq. 1
Np-237 (NP237)	Np-237	0.02	0.1	20				Eq. 1	Eq. 1
Tritium (H3)	H-3	20 dpm/ml		5				10 dpm/ml	
Sr-total (SR)	Sr (sum Sr-89 + Sr-90)	10		20				5	
Sr-90 (SR90)[c]	Sr-90	10		30				5	
Gamma Spectroscopy (ISPEC)	K-40, Cs-137 + Others(d)	See Table B-5		20				Eq. 1	
Gamma Spectroscopy (LEPD)	Am-241	5		20				Eq. 1	
U-nat (U)	Elemental U	0.06 µg/sample	0.3 µg/sample	20				0.2 µg/sample	0.2
U-236 (U 236)	U-236	140 pg/sample(h)		20				70 pg/sample	
U-238 (U 238)	U-238	0.06 µg/sample	0.3 µg/sample	20				0.2 µg/sample	
Sequential Analyses:									
Pu(∝) Iso and Sr-total (IPS)	As for individual analyses	As for individual analyses		25	As for individual analyses				
Pu(∝) Iso, Am-241 (IPA)				25					
Pu(∝) Iso, Am-241, Sr-total (IPSA)				25					
Pu(∝) Iso, U-nat (IUPU)				25					
Actinide(∝) Isotopic (ITPAC)(e)				25					
Cm(∝) Iso, Am-241(ICA)	Cm-242, Cm-244, Am-241(b)			20					
Pu(∝) Iso and U ISO (IPIU)				25					

(a) Time allowed following determination of results to receipt of results by Battelle.

(b) Report measured activity for Cm-246, and Cm-248 upon request of the Battelle Technical Administrator.

(c) If total Strontium is less than 15 dpm, Y ingrowth is not required.

(d) Report all isotopes present at levels exceeding Equation 5. If ordered by the Battelle Technical Administrator, report results for radionuclides in Table B-5 specified in the processing

instruction, regardless of the activity measured.

(e) Pu (α) Isotopic, Am-241, and Cm (α) Isotopic.

(f) 0.16 dpm for U-234, 0.15 dpm for U-238, and the greater of 0.007dpm and Equation 5 for U-235.

(g) Oral report required only when analytical results exceed level specified.

(h) CL is for U-236 in the presence of 0.2 microgram U-238 and 0.0014 microgram U-235.

Eq. 1 L_c=2(combined standard uncertainty)

TABLE 2. Number and Category of Bioassay Samples Analyzed

Procedure	FOURTH CONTRACT YEAR - GEL 4/1/08 through 3/31/09				FIFTH CONTRACT YEAR - GEL 4/1/09 through 3/31/10			
Code[a]	Total	IDP QC	%	GEL QC[b]	Total	IDP QC	%	GEL QC[b]
Urine								
H3	664	0	--	178	1285	0	--	388
SR90, SR	282	0	--	522	406	0	--	882
C14	--	0	--	--	--	0	--	--
AM241	96	0	--	402	158	0	--	701
AM243	70	7	10	66	103	6	6%	108
U235	--	0	--	--	--	0	--	--
ICM	8	0	--		25	0	--	
IPU	1406	0	--	1065	1730	2	0%	1742
IPUL	2	0	--	--		0	--	--
IPA	445	0	--	N/A	596	0	--	N/A
IPS	722	0	--	N/A	925	0	--	N/A
IPSA	172	25	15	N/A	323	23	7%	N/A
IPSR	--	0	--	--	--	0	--	--
ISPEC	--	0	--	--	--	0	--	--
ITPAC	220	0	--	N/A	271	0	--	N/A
ITH	3	0	--	6	21	0	--	48
IUPU	93	0	--	N/A	127	0	--	N/A
IPIU	10	0	--	N/A	38	0	--	N/A
IU	467	9	2	189	726	10	1%	465
NP237	7	0	--	--	13	0	--	24
U236	20	15	75	8	9	0	--	17
U mass	293	6	2	180	1314	17	1%	709
LEPD	--	0	--	--	--	0	--	--
PU241	--	0	--	--	--	0	--	--
Total	*4980*	*62*	*1*	*2616*	*8070*	*58*	*1*	*5084*
Fecal [c]								
U232	--	0	--	--	--	0	--	--
ICM	--	0	--	--	--	0	--	--
IU	--	0	--	--	--	0	--	--
AM241	--	0	--	79	2	0	--	88
IPU	1	0	--	82	1	0	--	88
IPA	41	6	15	N/A	57	10	18%	N/A
Total	*42*	*6*	*14*	*161*	*60*	*10*	*17*	*176*

[a] Procedures not specifically tested are evaluated with isotopic results from other procedures.

[b] N/A = not available. QC samples are tracked as isotopic analyses not as multiple analyses.

[c] Analyses not analyzed (IPUBA, IRA, ITPAC, IUPU, UNAT, IU, AM243)

Table 2 presents a breakdown of the numbers and categories for all bioassay samples analyzed, including personnel and QC samples. From 62 urine and 6 fecal QC samples submitted by IDP to GEL during the reporting period, GEL reported 4980 analytical urine results for 13 different analytes and 42 fecal results for 3 different analytes. The 68 QC samples represent 1.3% of the total analyses performed by GEL. In addition to these samples, GEL analyzed 6022 internal QC samples. The QC samples analyzed equaled 33% of the samples analyzed by GEL under their contract with Battelle.

GEL's performance was checked by determining detection level, bias, and precision based on the results of blank and spiked samples. Spiked samples fell into two categories: those spiked near the CL and those spiked at equal to or greater than three times the CL. These two categories were necessary to check compliance with the criteria for relative precision (S_B) specified by the Statement of Work. Satisfying these two categories also verified that GEL could detect sample activities near the CL.

DETECTION LEVELS

Various mathematical expressions and terminology can be used to describe a detection level. The statistical approach specified in the Statement of Work basically follows that of Currie (1968) and HPS N13.30 (HPS 1996). However, the HPS N13.30 formulas were modified to account for the difference between a priori estimates of detection levels based on counts (Currie 1968) and a posteriori estimates based on total activity, where chemical yield is determined specifically for each sample.

Two test criteria were used: the decision level (L_c) and the MDA (also called the detection level). The decision level was defined in the Statement of Work as the quantity of radioactivity or mass above which there is at least 95% confidence that the sample is not a blank (Type I error). If the measured value was greater than the L_c, the sample was considered likely to contain the radionuclide of interest. If the measured value was less than L_c, then the result was considered indistinguishable from a blank. The L_c was determined solely by measuring blank samples. Before the L_c was calculated, results that were significant outliers were eliminated from the data set. Outliers were identified by the use of the criteria of ASTM E178-94 (ASTM 1994).

Mathematically, L_c is defined by the following equation:

$$L_c = 2s_A$$

where, s_A equals the combined standard uncertainty of the net analyte reported.

The MDA was based on a 95% probability of detecting activity when the actual activity is equal to the MDA, and conversely a 5% probability of the results falling below the L_c and being judged to contain no activity (Type II error). The MDA, expressed in units of disintegrations per minute, is calculated from the same set of blanks as the L_c (outliers excluded), using the following equation:

$$MDA = \overline{X_o} + 2(t_{n-1})\ s_o + \frac{(t_{n-1})^2}{ERT}$$

Where

$\overline{X_o}$ = mean net result for the replicate blank samples, in disintegrations per minute

n = number of replicate blank measurements

(t_{n-1}) = the 95th quantile of the "student-t" distribution with (n-1) degrees of freedom

s_o = standard deviation of the net blank, in units of disintegrations per minute

E = the typical counter detection efficiency in counts per disintegration

R = the average fractional chemical recovery or yield

T = the typical counting time.

The above equation is considered appropriate for use with replicate blank results and for comparison with the equation in the contract statement of work, which is calculated with mean count data. In keeping with the philosophy of HPS N13.30, if t^2 is less than 3, then 3 is used instead. For uranium mass analyses, the analytical method does not produce count data; the unit for the analysis result and MDA is micrograms. Thus, the "3" term is not an appropriate part of the equation for the uranium mass analysis.

The present contract with GEL, specifies an operational year that ends March 31st, each year. This QC report covers the fifth operational year of contract 11530 effective date ending 2/23/10 and contract 112512 effective date beginning 2/24/10. It includes samples analyzed by GEL during period of April 1, 2009 through March 31, 2010.

The MDA values GEL calculates for their QC reports are based on mean values for parameters of equation 2 of the contract statement of work, and not replicate measurements. GEL also uses synthetic samples, whereas IDP uses real fecal and urine samples.

The IDP QC samples were evaluated by first calculating the L_c from blank samples, excluding outliers. This L_c was compared with the L_c calculated from GEL's own QC samples. Then, the MDA was calculated and compared with the CL and the MDA calculated from GEL's own QC samples. Values used for E, R, and T in the MDA equation were obtained from the laboratory, they are listed in Table 3. Finally, the percentage of QC samples spiked at the CL that were measured by the laboratory as having less than the decision level (i.e., no activity was detected) was determined, this percentage was then compared with the 5% allowed in the Statement of Work. Outliers were included in this test.

BIAS

Relative bias is defined as the mean fractional deviation of the reported results from the true values of spikes added to the samples. The formulas in the Statement of Work used to measure bias in sample results are the same as those in HPS N13.30 (1996). The mean relative bias, Br, is determined using:

$$B_r = \sum_{i=1}^{m} \sum_{j=1}^{n} \frac{B_{rij}}{N}$$

where n = number of spike samples in each level

m = number of spike levels

N = total number of spiked samples

B_{rij} = bias of a single measurement, defined as:

$$B_{rij} = \frac{(A_{ij} - A_{ai})}{A_{ai}}$$

where A_{ij} = the jth measured value of the ith spike level,
A_{ai} = the true value of the ith spike level

TABLE 3. Typical Chemical Yield (R), Typical Detector Efficiencies (E), and Counting Time (T) Values from GEL Quality Control Report

Matrix	Nuclide/ Method	Count Minutes	*Contract Limit*[a]	Counter Efficiency 2008-2009	Counter Efficiency 2009-2010	Chemical Yield 2008-2009	Chemical Yield 2009-2010
Urine	^{3}H	20	*20*	0.243	0.243	N/A	N/A
	Total Sr	60	*10*	0.379	0.379	0.757	0.778
	SR90	60	*10*	0.379	0.379	0.76	0.759
	^{241}Am	2520	*0.02*	0.391	0.391	0.8175	0.867
	^{243}Am	2520	*0.02*	0.391	0.391	0.8668	0.922
	$^{242}Cm/^{244}Cm$	2520	*0.02*	0.391	0.391	0.8175	0.867
	^{237}Np	2520	*0.02*	---	0.391	---	0.717
	$^{239}Pu/^{238}Pu$	2520	*0.02*	0.391	0.391	0.925	0.902
	IPUL	10000	*0.005*	---	---	---	---
	$^{228}Th/^{230}Th/^{232}Th$	2520	*0.1*	0.386	0.386	0.9156	0.900
	$^{234}U/^{235}U/^{238}U$	2520	*0.02*	0.386	0.386	0.9047	0.862
	Uranium	--	*0.06*	N/A	N/A	N/A	N/A
Fecal	^{241}Am	960	*0.8*	0.391	0.391	0.909	8.865
	$^{238}Pu/^{239}Pu$	960	*0.2*	0.391	0.391	0.914	0.801

(a) Units dpm/sample except dpm/mL for ^{3}H, and µg/sample for U.

(b) Only one sample analyzed

(c) NA = Not available. No samples completed.

Outliers were excluded from the test, but not ignored for the procedure evaluation. As stipulated in the Statement of Work, the mean relative bias shall fall within ± 20% when calculated from 15 to 50 spiked samples, and within ± 10% when calculated from over 50 samples.

PRECISION

The precision statistic used for this contract was S_B from HPS N13.30 (1996), but the limits differ from that standard. S_B is given by:
where the symbols are the same as for relative bias (B_r).

The above equation is valid for samples spiked at one or more levels, subject to the limits

$$S_B = \sqrt{\sum_{i=1}^{m}\sum_{j=1}^{n} \frac{(B_{rij} - B_r)^2}{(N-1)}}$$

for the relative precision, which depend on the activity of the spikes relative to the CL. Specifically, the relative precision statistics shall be less than or equal to 0.4 for samples spiked greater than three times the CL and less than or equal to 0.5 for samples spiked between one and three times the CL. Outliers were not included in the determination of precision.

FINDINGS

Results from three types of QC samples were available: 1) those prepared by GEL and analyzed as single-blinds (spike amount unknown to the analyst), 2) those submitted by IDP and analyzed as single-blinds (spike amount unknown to the analyst), and 3) those submitted by IDP and analyzed as double-blinds (spike amount and sample origin unknown to the analyst).

Single-blind samples this year included 10 urine samples prepared by RPG for isotopic uranium analysis. Because a depleted uranium source is used to spike the samples, they are run as single-blinds. The remaining 44 audit samples submitted by IDP were double-blind samples and included 10 actual fecal samples. The results of the statistical tests (see Table 4 and Appendix A) are discussed below. Statistical results from the present and previous years are compared in Table 5.

OUTLIERS

Analytical results that are biased by "blunders" during the analysis, should not be included in the data set used for the statistical evaluation of the analytical procedure, but too many outliers would indicate poor laboratory performance (see Table 6). GEL (see Appendix B) identified some outliers associated with their laboratory control samples (blanks and spiked). GEL declared two ^{243}Am urine data points and three ^{241}Am fecal data points as outliers, because the results were erroneous due to laboratory error they were excluded from the database.

TABLE 4. Summary of Statistical Values by Nuclide

	Sample	Blank (dpm)				Spike level at CL (dpm)			Spike Level > 2CL (dpm)		
Isotope[a]	Source	n	L_c	MDA	**CL**	n	B_r	S_B	n	B_r	S_B
^{3}H(dpm/mL)	**IDP**	0	…	…	**20**	0	…	…	0	…	…
	GEL	191	0.5371	0.826	**20**	191	0.02	0.08	0	…	…
^{14}C (dpm/ml)	**IDP**	0	…	…	**10**	0	…	…	0	…	…
Total Sr/^{90}Sr	**IDP**	0	…	…	**10**	23	-0.045	0.12	0	…	…
	GEL	291	0.76	4.44	**10**	290	0.02	0.10	290	0.02	0.09
^{237}Np	GEL	8	0.01	0.01	**10**	8	0.16	0.21	8	0.090	0.054
^{228}Th	GEL	16	0.026	0.040	**0.1**	0	…	…	0	…	…
^{229}Th	GEL	15	0.008	0.015	**0.1**	0	…	…	0	…	…
^{232}Th	GEL	16	0.007	0.015	**0.1**	16	0.02	0.10	16	0.040	0.07
^{230}Th	GEL	16	0.018	0.031	**0.1**	0	…	…	0	…	…
^{242}Cm	GEL	84	0.004	0.010	**0.02**	0	…	…	0	…	…
^{243,244}Cm	GEL	232	0.004	0.011	**0.02**	80	0.12 (e)	0.28	81	0.014	0.087
^{238}Pu-urine	**IDP**	25	0.004	0.011	**0.02**	0	…	…	0	…	…
	GEL	570	0.004	0.010	**0.02**	0	…	…	569	-0.03	0.08
feces	**IDP**	5	0.011	0.036	**0.2**	5	0.08	0.19	0	…	…
	GEL	22	0.01	0.050	**0.2**	0	…	…	0	…	…
^{239,240}Pu-urine	**IDP**	0	…	…	**0.02**	20	0.09	0.20	5	0.007	0.062
	GEL	570	0.004	0.011	**0.02**	569	0.01	0.25	0	…	…
feces	**IDP**	5	0.015	0.044	**0.2**	5	-0.02	0.13	0	…	…
	GEL	22	0.01	0.054	**0.2**	19	0.07	0.25	19	-0.021	0.053
^{241}Am-urine	**IDP**	0	…	…	**0.02**	18	-0.06	0.21	5	0.104	0.209
	GEL	232	0.004	0.100	**0.02**	231	0.068	0.29	232	-0.021	0.099
feces	**IDP**	5	0.051	0.116	**0.2**	5	0.04	0.08	0	…	…
	GEL	22	0.01	0.048	**0.2**	19	0.18	0.23	19	-0.015	0.094
^{243}Am-urine	**IDP**	6	0.004	0.013	**0.02**	0	…	…	0	…	…
	GEL	36	0.006	0.013	**0.02**	36	-0.05	0.23	36	0.02	0.09
^{233,234}U	**IDP**	0	…	…	**0.02**	0	…	…	0	…	…
	GEL	149	0.009	0.021(e)	**0.02**	0	…	…	0	…	…
^{235,236}U	**IDP**	10	0.00	0.012	**0.02**	0	…	…	0	…	…
	GEL	149	0.006	0.013	**0.02**	0	…	…	0	…	…
^{238}U	**IDP**	0	…	…	**0.02**	0	…	…	10	0.02	0.09
	GEL	149	0.008	0.020	**0.02**	147	0.11 (e)	0.41 (e)	147	-0.03	0.11
^{236}U-ICPMS[b]	**IDP**	0	…	…	**140 pg**	17	-0.02	0.32	0	…	…
	GEL	5	26.5	26.5	**140 pg**	4	0.00	0.02	4.00	-0.02	0.02
^{238}U-ICPMS[b]	**IDP**	0	…	…	**0.06 µg**	0	…	…	17	-0.02	0.32
	GEL	151	0.014	0.01	**0.06 µg**	102	0.06	0.12	183	0.002	0.079

(a) Analyzed in urine matrix unless otherwise noted.
(b) Units for performance indicators are the same as the units for CL.
(c) Failed performance criterion.
(d) Possible environmental contaminant.
(e) Within statistical uncertainty
(f) Stats for Cm same as Am-241

TABLE 5. Comparison of Quality Control Statistics Between the Third and Fourth Contract Year with GEL Using QC Samples Submitted by IDP

		Report	Blanks			Spike Level at CL			Spike Level at > 3CL		
Nuclide	CL	Year	n	L_c	MDA	n	B_r	S_B	n	B_r	S_B
^{3}H	20 dpm/mL	2009	0	...	...	0	...	...	0	...	...
		2008	0	...	...	0	...	...	0	...	...
Sr	10 dpm	2009	0	...	...	23	0	0	0	...	...
		2008	6	1.0586	2.3531	19	-0.062	0.106	0	...	...
U	0.06 mg	2009	0	...	...	0	...	...	17	-0.02	0.32
(ICPMS)		2008	0	...	...	0	...	...	6	-0.19	0.25
^{235}U	0.02 dpm	2009	10	0.004	0.012	0	...	...	0	...	...
		2008	9	N/A	0.014	0	...	...	0	...	...
^{238}U	0.02 dpm	2009	0	...	...	0	...	...	10	0	0
		2008	0	...	...	0	...	...	9	-0.07	0.08
^{238}Pu	0.02 dpm	2009	25	0.004	0.011	0	...	...	0	...	...
(urine)		2008	25	0.003	0.0089	0	...	...	0	...	...
^{238}Pu	0.2 dpm	2009	5	0.011	0.036	5	0.080	0.187	0.00	...	...
(fecal)		2008				6	0.1264	0.504(c.)			
^{239}Pu	0.02 dpm	2009	0	...	...	20	0.092	0.200	5	0.007	0.062
(urine)		2008	5	0.0057	0.0164	20	-0.04	0.32	0	...	...
^{239}Pu	0.2 dpm	2009	5	0.01	0.04	5	-0.02	0.13	0	...	...
(fecal)		2008	0	...	...	6	-0.04	0.16	0	...	...
^{241}Am	0.02 dpm	2009	0	...	...	18	-0.058	0.212	5	0.104	0.209
(urine)		2008	6	0.0014	0.0078	14	0.18	0.32	0	...	...
^{241}Am	0.2 dpm	2009	5	0.051	0.116	5	0.037	0.080	0	...	...
(fecal)		2008	0	...	...	6.0	-0.03	0.06	0	...	...
^{243}Am	0.02 dpm	2009	6	0.004	0.013	0	...	...	0	...	...
		2008	7	0.005	0.015	0	...	...	0	...	...

Note: L_c and MDA units same as CL. B_r and S_B are unitless (fractional values).

TABLE 6. Other Indicators of Analytical Uncertainty (IDP Samples)

Nuclide	IDP QC Samples Analyses	IDP QC Samples Outliers	Performance Evaluation Samples Spikes at CDL IDP	Spikes at CDL GEL	False Negatives (%) IDP	False Negatives (%) GEL	Analytical Samples 2009-2010 Yield Flags	Analytical Samples 2009-2010 Failed Analyses
Urine								
^{3}H	0	0 (0)	0	191		0 (0)		0.1%
Sr	23	0 (0)	23	290	0 (0)	0 (0)	3.0%	2.1%
^{235}U	10	0 (0)	0	0			1.9%	
^{238}U[(a)]	17	0 (0)	10	147	0 (0)	0 (0)		
^{238}Pu	25	0 (0)	0	0			1.5%	0.6%
^{239}Pu	25	0 (0)	20	569	0 (0)	1 (0.2%)	1.5%	0.6%
^{241}Am	23	0 (0)	18	231	0 (0)	0 (0)	1.2%	0.9%
^{243}Am	22	0 (0)	0	36			3.3%	3.3%
U mass [(a)]	17	0 (0)	17	183	0 (0)	0 (0)		
Total	*162*		*88*	*1647*				
Feces								
^{241}Am	10	0 (0)	5 (a)	19	0 (0)	0 (0)		2%
^{238}Pu	10	0 (0)	5 (a)	0	0 (0)	0 (0)	6%	2%
^{239}Pu	10	0 (0)	5 (a)	19	0 (0)	0 (0)	6%	2%
Total	*30*		*15*	*38*				

(a) sample spiked at >3 CL

TRITIUM

Effective June 2006, the tritium intercomparison program by IDP was discontinued, performance indicators will be evaluated through GEL's QC program. The control samples run by GEL also met all the acceptance criteria tested as part of the quality control program. The tritium analyses were considered acceptable.

STRONTIUM-90 AND TOTAL STRONTIUM

The total strontium procedure is used to screen samples to determine which will require analysis for ^{90}Sr. Samples with total strontium results less than 15 dpm do not undergo further analysis. Samples with results greater than or equal to 15 dpm may undergo ^{90}Y in growth to specifically determine ^{90}Sr levels. The calculated MDA, reported by GEL and tested by IDP, for the total strontium part of the analysis was less than 55% of the CL. The relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all within limits. The 23 samples spiked at the contractual level by IDP were all detected. The strontium urinalysis procedure was concluded to be acceptable. There was an issue however, in September 2009 with a batch of samples to be analyzed for strontium that were

switched with another batch. This is discussed further in the section on follow-up concerns and documentation of the investigation provided in Attachment 2.

PLUTONIUM-238 AND –239

Samples spiked with ^{238}Pu and ^{239}Pu were analyzed using the same procedures and same reagents. The two isotopes are differentiated only at the end of the procedure by alpha spectrometry. Therefore, laboratory performance is expected to be similar for both isotopes using any of the seven procedures that incorporate plutonium analysis (IPU, IPA, IPS, IPSA, IPSR, IUPU, and ITPAC).

The MDAs and performance statistics for ^{239}Pu and ^{238}Pu in urine were acceptable. The 20 samples spiked at the CL and the five spiked at greater than three times the CL for ^{239}Pu were reported and all showed detection greater than the decision level. There were 25 blank samples analyzed for ^{238}Pu activity, none of the 25 samples detected activity in excess of the decision level. Overall the plutonium urinalyses were considered acceptable.

The MDA and performance statistics for ^{239}Pu and ^{238}Pu in feces were acceptable. Approximately 15% of the fecal samples analyzed were duplicated to test the consistency of the aliquoting procedure. A review of the duplicate samples determined that the aliquoting procedure produced results within 3 sigma of the initial results. The fecal aliquoting procedure was acceptable. This year IDP submitted 5 actual fecal samples spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The precision and bias for ^{239}Pu and ^{238}Pu met the performance criteria. The performance statistics reported by GEL for ^{239}Pu met the acceptance criterion. The low-yield rate and failed analysis rate for fecal sampling over the contract year was 6% and 2% respectively, which was below the contractual level of 10%. The problem of technician errors resulting in a failed analysis rate greater than 10% seems to have been corrected. Overall the plutonium fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

The four tissue samples submitted for ^{238}Pu, ^{239}Pu, ^{241}Am and ^{241}Pu analysis, showed results consisted with direct measurements. The analysis of the tissue samples not only tested skin tissue analysis but also the expedite processing, see Attachment A for details.

ISOTOPIC URANIUM

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, ^{233,234}U was not evaluated. IDP submitted 10 urinalyses samples throughout the year for isotopic uranium analysis. The performance statistics for ^{235}U and ^{238}U were reviewed and the MDA for ^{235}U and the bias and precision for ^{238}U were acceptable.

URANIUM MASS

No concerns were identified with the uranium mass (^{238}U-ICPMS) urinalysis program and it was considered acceptable. Because IDP uses a 0.2 µg screening level for elemental uranium, samples spiked at 0.06 µg were discontinued. The MDA at the contractual level of 0.06 µg was evaluated through GEL's program and was found to be acceptable. The relative bias and precision were likewise acceptable. The bias and precision from the 17 samples submitted IDP met the acceptance criteria. The bias and precision was tested by IDP at 0.2 µg and by GEL at 1 µg/L and at 0.05/L µg.

In February 2009, the KPA uranium mass analysis was being phased out and replaced with the ICPMS analysis for ^{238}U, which comprises 99% of the uranium isotopic mixture by mass. During the first quarter GEL was processing both elemental uranium via KPA and ICPMS for ^{238}U. Sample results reported in the first quarter for ^{238}U via ICPMS initially were reported without a corresponding analyzed volume. Because GEL reported the analyzed volume in liters

and the database rounds to 2 significant figures, the resulting 0.001 L analyzed volume was rounded to 0.00 L. GEL changed their programming to reflect milliliters rather than liters. Also, effective June 1, 2009, GEL changed the analyzed volume from 1 ml to 2 ml in order to more comfortably comply with the CL of 0.06 µg/sample.

URANIUM-236 VIA INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICPMS)

A new ^{236}U analysis procedure was initiated in June 2007 and the procedure was formally approved in June 2008. The analysis for ^{236}U uses inductively coupled plasma mass spectrometry. IDP submitted ten blank samples and the MDA was found to be acceptable. The MDA and relative bias and precision reported by GEL met the performance criteria. The ^{236}U analysis procedure was considered acceptable.

AMERICIUM-241

The ^{241}Am fecal and urine analysis met the acceptance criteria for MDA, relative bias and precision. The MDA as reported by GEL and tested by IDP was less than 50% of the contractual level. All 18 of the ^{241}Am samples spiked at the contractual detection level (CL) and the 5 spiked at greater than three times the CL were detected. The relative bias and precision as reported by GEL and tested by IDP met the performance criteria. The current AM241 urinalysis procedure was considered acceptable.

The ^{241}Am fecal duplicate samples were evaluated and it was concluded that the aliquoting procedure produced results within the control limits. This year IDP submitted 5 blank fecal samples and 5 fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. The failed analysis rate for ^{241}Am fecal analyses was 2%, which was below the contractual level of 10%. The problem of technician errors resulting in a failed analysis rate greater than 10% seems to have been corrected. Overall the ^{241}Am fecal analyses were considered acceptable.

The four tissue samples submitted for ^{238}Pu, ^{239}Pu, ^{241}Am and ^{241}Pu analysis, showed results consisted with direct measurements. The analysis of the tissue samples not only tested skin tissue analysis but also the expedite processing, see Attachment A for details.

AMERICIUM-243

The AM243 procedure was identical to the AM241 procedure, except a different tracer is used (^{244}Cm instead of ^{243}Am). The seven blank ^{243}Am QC samples submitted were all reported with results less than the decision level and the calculated MDA was 35% of the contractual detection level. The performance statistics for ^{243}Am, as tested by GEL, met the acceptance criteria. The ^{243}Am procedure was concluded to be acceptable.

ISOTOPIC CURIUM

IDP did not submit QC samples to test the isotopic curium program, therefore performance statistics were based on the GEL QC results. GEL tested the MDA for ^{242}Cm and ^{244}Cm and the relative bias and precision for ^{244}Cm. The results met the acceptance criteria and the isotopic curium urinalysis program was considered acceptable.

ISOTOPIC THORIUM

IDP also did not submit QC samples to test the isotopic thorium program, therefore performance statistics were based on the GEL QC results. GEL tested the MDA for ^{228}Th, ^{229}Th, ^{230}Th and ^{232}Th and the relative bias and precision for ^{232}Th. The results met the acceptance criteria and the isotopic thorium urinalysis program was considered acceptable.

FOLLOW-UP ON CONCERNS DURING THE FIFTH CONTRACT YEAR

The main emphasis during the first part of the fourth contract year was developing an ICPMS procedure for ^{236}U analysis. This was accomplished in June 2008. There were a few concerns carried over from the fourth contract year, primarily technician errors involving sample batches, typically consisting of loss of sample, cross contamination, forgetting to perform a task, or lack of proper documentation. The largest concern during the fifth contract year was laboratory errors resulting in failed analyses or incorrectly reporting of data. Over the contract year there were 36 samples whose results were not reported due to laboratory errors and 20 samples with incorrect results reported. A review by IDP of the yield rate and failed analysis rate was greater than 10% during the first quarter but corrective actions implemented by GEL seemed to have corrected the concerns and by the end of the year the low yield and failed analyses rates were acceptable.

In the second quarter there was one event resulting in two batches, each with nine samples, being switched. This resulted in incorrectly reporting the strontium results for 18 workers, all of whom needed to be re-sampled. The incident is documented by IDP in Attachment 1 as well as in Appendix B, in the Incident Report Section under work order 234742. The mix-up occurred when the filters with the sample precipitates for two separate batches were manually labeled with identical batch numbers. Without consulting the Group Leader or Senior Analyst, the technician who labeled the planchettes determined incorrectly which group had the wrong batch labels. The corrective actions that GEL implemented included generating LIMS labels and affixing them to the petri dishes holding the counting filters, documenting all issues with the batches pro provide information for data reviewers/validators and decisions on vague situations will be resolved by the group leader or senior staff.

In December there were two sample planchettes that were loaded in the detectors in the wrong order resulting in the results being associated with the wrong planchettes, effectively the samples were switched. The planchettes were being analyzed for the presence of plutonium. Because the error was not a result of incorrectly labeling a planchete, the corrective actions from the strontium event did not apply. The planchettes were loaded in the wrong detectors because confusion with the numerical order from multiple sample ID's. GEL's corrective action included recounting the samples in the correct detectors and developing a program in LIMS to re-order samples with multiple sample IDs to prevent confusion when IDs are not in numerical order.

In both the September and December incident involving switched samples and incorrectly reporting of sample data, the errors were identified through the IDP's double-blind QC program. To verify whether other samples had been switched but not identified, IDP reviewed the records since the contract with GEL was initiated in 2005. Out of approximately 50,000 results there were 318 results that exceeded the screening levels and of those, only 38 were not associated with a known intake. The 38 results were reviewed and all but 6 were explained. The 6 samples all had been previously investigated and there was no evidence of them having been switched. The 6 samples with unresolved high results reflects only 0.012% of the total samples processed since 2005.

SUMMARY OF THE BIOASSAY QUALITY CONTROL REPORT FROM GEL INCORPORATED, FOR THE CONTRACT 11530 FOURTH YEAR 2006/2007[a]

GEL reported all analytical batches were analyzed with a reagent blank (U mass only), matrix blank or both. GEL considered blanks in control when the calculate MDA was less than the Contract Limit (CL) and the L_c was less than ½ CL (see Appendix B). In addition, the chemical tracer yields were evaluated against the yield requirements stated in the subject contract. Overall, GEL believed that the blank and spike data for each analytical process demonstrated that the analyses were in control.

In the review GEL indentified laboratory control samples that had yields greater than 125% as well as one excreta sample that had a tracer yield greater than 125%. GEL also indentified laboratory control samples that met the criteria for low yield, but likewise a review of excreta sample results found the low yield rate to be acceptable.

RESULTS FROM INTERCOMPARISON PROGRAMS

GEL participated in two intercomparison programs (Appendix C – Intercomparison Programs) in the fourth contract year. Between September and October 2009, GEL participated in the National Institute of Standards and Technology's program testing the relative bias and precision for ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{243}Cm,^{238}Pu, ^{239}Pu, ^{241}Am, ^{230}Th, ^{235}U, ^{238}U, ^{234}U and ^{90}Sr in synthetic feces. GEL met the acceptance criteria for relative bias and precision for all isotopes except. GEL also participated in the National Institute of Standards and Technology's program testing the relative bias and precision for ^{241}AM+^{243}CM, ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{238}Pu, ^{240}Pu, ^{241}Am, ^{230}Th, ^{235}U, ^{238}U, ^{234}U and ^{90}Sr, in synthetic urine. GEL met the acceptance criteria for relative bias and precision on all isotopes.

(a) Summaries are taken from GEL (2009).

REFERENCES

American Society for Testing and Materials (ASTM). 1994. *Standard Practice for Dealing with Outlying Observations*. ASTM E 178-94, ASTM, Philadelphia, Pennsylvania.

Health Physics Society (HPS). 1996. *Health Physics Society Standard for Performance Criteria for Radiobioassay*. HPS N13.30-1996, HPS, McLean, VA.

Battelle. 2007. "Contract No. 11530 Mod 5 Bioassay Radiochemical Analytical Services" General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

Battelle. 2009. "Contract No. 11530 Mod 11 Bioassay Radiochemical Analytical Services" General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

Currie, L. A. 1968. "Limits for Qualitative Detection and Quantitative Determination." *Analytical Chemistry* 40(3):586-593.

Pifer, S. 2009. " PNNL Annual QC Package, Second Contract Year 2006/2007 (April 1, 2006 – March 31, 2007)" Letter Report, General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

Pizarro, S. 2009. " PNNL Annual QC Package, Second Contract Year 2008/2009 (April 1, 2008 – March 31, 2009)" Letter Report, General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

Pizarro, S. 2010. " PNNL Annual QC Package, Second Contract Year 2009/2010 (April 1, 2009 – March 31, 2010)" Letter Report, General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

Remmington, Richard D. and M. Anthony Schork, 1970. *Statistics with Applications to the Biological and Health Sciences*. Printice-Hall. New York.

Miller, Irwin and John Freund, 1977. *Probability and Statistics for Engineers*. Prentice-Hall. Englewood Cliffs, New Jersey.

RELEVANT PROCEDURES AND CORRESPONDENCE

Antonio, C. L.. 2010. "Results of the PNL Excreta Bioassay Quality Control Oversight Program for April 1, 2009 through June 30, 2009." Letter Report, Pacific Northwest Laboratory, Richland, Washington. (Available from the Hanford Radiological Records Historical File.)

Antonio, C. L. 2010. "Results of the PNL Excreta Bioassay Quality Control Oversight Program for July 1, 2009 through September 30, 2009." Letter Report, Pacific Northwest Laboratory, Richland, Washington. (Available from the Hanford Radiological Records Historical File.)

ATTACHMENT 1: TISSUE ANALYSES

Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Wednesday, July 14, 2010 4:13 PM
To: Antonio, Cheryl L
Subject: FW: Tissue specimen results

Is this what you were thinking of?

Gene Carbaugh
Staff Scientist and Internal Dosimetry Manager
Pacific Northwest National Laboratory

From: Carbaugh, Eugene H
Sent: Monday, September 21, 2009 9:22 AM
To: MacLellan, Jay; Antonio, Cheryl L; Lynch, Timothy P (PNNL)
Subject: Tissue specimen results

GEL sent us the priority tissue specimen analyses this morning. The attached Excel file compiles the GEL results and Tim's preliminary in vivo results. Pretty good comparison. Will be substantially better when Tim provides the corrected in vivo Pu-alpha estimates based on the x-rays. His preliminary results didn't subtract out the Am-241 contribution thus are biased high.

Wound Count Tests.xls

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Tissue Specimen Radiochemical Analysis by GEL

All tissue samples submitted 9/14/2009 for analysis

Tissue	46				48			
Date	2/7/1985				2/7/1985			
Method	Radiochem	Radiochem	In Vivo	Ratio RC/InV	Radiochem	Radiochem	In Vivo	Ratio RC/InV
Process	E	E			E	E		
Units	dpm	nCi	nCi		dpm	nCi	nCi	
Pu-239	3.53E+04	15.90			502	0.226		
Pu-238	781	0.35			10.5	0.00473		
Pu-alpha	3.61E+04	16.25	112	6.89	512.5	0.231	2.6	11.26
Am-241	6.11E+03	2.75	6.8	2.47	82.1	0.0370	0.07	1.89
Pu-241	n.a.				n.a.			
Pu-α/Am	5.91	5.91	16.47		6.24	6.24	37.14	
Turnaround	24 h				24 h			

Tissue	45				47			
Date	1/29/1985				2/7/1985			
Method	Radiochem				Radiochem			
Process	P				P			
Pu-239	4.33E+04	19.50			5.91E+03	2.66		
Pu-238	1.03E+03	0.46			1.44E+02	0.06		
Pu-alpha	4.43E+04	19.97	92	4.61	6.05E+03	2.73	8.5	3.12
Am-241	7.30E+03	3.29	5.5	1.67	6.84E+02	0.31	0.5	1.62
Pu-241	9.86E+04	44.41			1.44E+04	6.49		
Turnaround	7 d				7 d			

Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Wednesday, July 14, 2010 4:21 PM
To: Antonio, Cheryl L
Subject: Activity Ratios from 1985 analyses of tissue specimens

You can compare these (from my paper in publication) with the ratios of last fall's analyses

MATERIAL COMPOSITION

Isotopic composition of the wound contamination was estimated based on tissue specimens collected from the two surgeries. One of the larger tissues excised from each surgery underwent radiochemical analysis. The results, shown in Table 2, indicated that there was a distinct difference in the activity ratios at the wound site between day 0 and day 7. This discrepancy led to independent analysis by two laboratories of a second tissue sample from the day 7 surgery. The results showed good agreement for the alpha activity ratios, leading to the conclusion that the difference in composition between the two dates was real and might be accounted for by differential transport from the wound site for some of the initially deposited ^{241}Am. The urine data appeared to support that contention, as evidenced by more ^{241}Am being excreted than $^{239+240}$Pu in the first 24 h following the wound and the observation of a gradual increase in the $^{239+240}$Pu:^{241}Am ratio in urine over the first 100 d. Lab A also acknowledged some problems with its ^{241}Pu analysis. The mean of the two Lab B analyses was used as the best estimate of activity relationships at day 7.

Table 2. Activity ratios in excised tissue specimens.

	Day 0 Tissue	Day 7 Tissue 9	Day 7 Tissue 10	
Activity Ratio	Lab A	Lab B	Lab A	Lab B
$^{239+240}$Pu:^{241}Am	5.9	12	11	11
^{238}Pu:^{241}Am	0.12	0.37	0.25	0.23
^{241}Pu:^{241}Am	17	31	2.6	20
^{241}Pu:$^{239+240}$Pu	2.9	2.4	0.24	1.9

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Tissue Specimen Radiochemical Analysis by GEL

All tissue samples submitted 9/14/2009 for analysis

Tissue	46				48			
Date	2/7/1985				2/7/1985			
Method	Radiochem	Radiochem	In Vivo	Ratio RC/InV	Radiochem	Radiochem	In Vivo	Ratio RC/InV
Process	E	E			E	E		
Units	dpm	nCi	nCi		dpm	nCi	nCi	
Pu-239	3.53E+04	15.90			502	0.226		
Pu-238	781	0.35			10.5	0.00473		
Pu-alpha	3.61E+04	16.25	112	6.89	512.5	0.231	2.6	11.26
Am-241	6.11E+03	2.75	6.8	2.47	82.1	0.0370	0.07	1.89
Pu-241	n.a.				n.a.			
Pu-α/Am	5.91	5.91	16.47		6.24	6.24	37.14	
Turnaround	24 h				24 h			

Tissue	45				47			
Date	1/29/1985				2/7/1985			
Method	Radiochem				Radiochem			
Process	P				P			
Pu-239	4.33E+04	19.50			5.91E+03	2.66		
Pu-238	1.03E+03	0.46			1.44E+02	0.06		
Pu-alpha	4.43E+04	19.97	92	4.61	6.05E+03	2.73	8.5	3.12
Am-241	7.30E+03	3.29	5.5	1.67	6.84E+02	0.31	0.5	1.62
Pu-241	9.86E+04	44.41			1.44E+04	6.49		
Turnaround	7 d				7 d			

Antonio, Cheryl L

From: Stan Morton [stan.morton@gel.com]
Sent: Friday, September 04, 2009 1:21 PM
To: Carbaugh, Eugene H; MacLellan, Jay; Antonio, Cheryl L
Cc: 'April Rhineheart'; 'james westmoreland'; 'Bob Timm'
Subject: RE: TRIM: RE: TRIM: RE: TRIM: Tissue Samples

May we destroy these samples for the analyses?

stan

Stan Morton, Manager
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stan.morton@gel.com
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From: Carbaugh, Eugene H [mailto:gene.carbaugh@pnl.gov]
Sent: Friday, September 04, 2009 2:17 PM
To: Stan Morton; MacLellan, Jay; Antonio, Cheryl L
Cc: April Rhineheart; james westmoreland; Bob Timm
Subject: RE: TRIM: RE: TRIM: RE: TRIM: Tissue Samples

Stan, et al.,

Below are the estimated activities of each of the four tissue specimens. In a real-life situation, you could expect similar data from us at the time a tissue specimen analysis would be requested. The tissues are all from a 1985 wound incident and are each about the size of a finger-nail clipping. Each tissue is on a piece of cotton gauze, inside a labeled zip lock bag. Each bag is inside a nominal 100 ml plastic sample bottle. We counted the tissues by placing the zip lock bag in direct contact with the detector. There is no smearable activity on any of the bags.

	Am-241[(a)] (nCi)	Pu-α[(b)] (nCi)	Pu-241[(c)] (nCi)
Tissue 45	5.5	92	43
Tissue 46	6.8	112	54
Tissue 47	0.5	8.5	3.9
Tissue 48	0.07	2.6	0.55

(a) Based on 59.5 keV photon (Aug 2009)
(b) Based on 17 keV photons (Aug 2009)
(c) Calculated based on activity ratio from 1985 measurement and decay correction of 24-years

Gene Carbaugh, CHP

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From: Stan Morton [mailto:stan.morton@gel.com]
Sent: Friday, September 04, 2009 12:55 PM
To: MacLellan, Jay
Cc: 'April Rhineheart'; Carbaugh, Eugene H; 'james westmoreland'; 'Bob Timm'
Subject: RE: TRIM: RE: TRIM: RE: TRIM: Tissue Samples

Thank you.

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From: MacLellan, Jay [mailto:jay.maclellan@pnl.gov]
Sent: Friday, September 04, 2009 12:57 PM
To: Stan Morton
Cc: April Rhineheart; Carbaugh, Eugene H; james westmoreland; Bob Timm
Subject: RE: TRIM: RE: TRIM: RE: TRIM: Tissue Samples

We want to make some preliminary measurements using a new instrument that would be used in an actual emergency situation. Gene just said he will try to get that done by next Tuesday. I'd like to get them out by Thursday. I don't want anyone to have to work on the weekend. This is still preliminary, and we'll have better dates next week.

Jay MacLellan
509-376-7247
jay.maclellan@pnl.gov

From: Stan Morton [mailto:stan.morton@gel.com]
Sent: Friday, September 04, 2009 11:54 AM
To: MacLellan, Jay
Cc: 'April Rhineheart'; Carbaugh, Eugene H; 'james westmoreland'; 'Bob Timm'
Subject: TRIM: RE: TRIM: RE: TRIM: Tissue Samples

Jay,
We have evaluated a number of options, and are waiting for some additional information, but will be ready by next week. James is on travel today, but should be available this weekend. We will finalize the plans and be

ready next week. I know in an actual emergency, we would not have this advanced notice, but if you have an idea of the day the samples would be available, it would help for planning purposes.

Thanks,
stan

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From: MacLellan, Jay [mailto:jay.maclellan@pnl.gov]
Sent: Friday, September 04, 2009 12:44 PM
To: Stan Morton
Cc: April Rhineheart; Carbaugh, Eugene H
Subject: RE: TRIM: RE: TRIM: Tissue Samples

Stan,

What are your schedule limitations for the shipping? We don't want you to charter a special flight for this?

Jay MacLellan
509-376-7247
jay.maclellan@pnl.gov

From: Stan Morton [mailto:stan.morton@gel.com]
Sent: Friday, September 04, 2009 11:39 AM
To: MacLellan, Jay
Cc: 'April Rhineheart'; 'Dale Mori'; 'Bob Timm'; 'james westmoreland'; Carbaugh, Eugene H; Antonio, Cheryl L
Subject: TRIM: RE: TRIM: Tissue Samples

Thank you. We will look for them then.

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From: MacLellan, Jay [mailto:jay.maclellan@pnl.gov]
Sent: Friday, September 04, 2009 12:38 PM
To: Stan Morton
Cc: April Rhineheart; Dale Mori; Bob Timm; james westmoreland; Carbaugh, Eugene H; Antonio, Cheryl L
Subject: RE: TRIM: Tissue Samples

Yes, it is still in the works. You will probably get them next week, and I'll send details as soon as we know what they are.

Jay MacLellan
509-376-7247
jay.maclellan@pnl.gov

From: Stan Morton [mailto:stan.morton@gel.com]
Sent: Friday, September 04, 2009 11:33 AM
To: MacLellan, Jay
Cc: 'April Rhineheart'; 'Dale Mori'; 'Bob Timm'; 'james westmoreland'
Subject: TRIM: Tissue Samples

Jay,
Are you still planning to implement the Priority (9/15-day) IPUBA analysis and Emergency (24-hr) IPA analysis for the tissue samples?

Thanks,
Stan

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ATTACHMENT 2: SWITCHED STRONTIUM SAMPLE INVESTIGATION

Internal Dosimetry Quality Problem Report

Quality Problem Report: Strontium urine samples switched resulting in erroneously reporting sample results.

PNNL Assessment Tracking System (ATS) Entry:

Date discovered: 9/23/2009

Owner: Eugene H. Carbaugh

Condition:

On September 23, 2009 it was determined that two worker's baseline strontium urinalysis results were not consistent with their follow-up strontium urinalyses or with their work history. A third worker likewise had elevated strontium levels reported for their baseline sample, but follow-up samples and work history review did not rule out a potential prior to Hanford exposure. All three worker's baseline samples, dated July 29 or 30, 2009, were processed in a batch of nine samples. The baseline strontium results for the three workers were reported at 10.1 dpm, 9.5 dpm and 8.8 dpm. A data check by the analytical lab, of the July 2009 worker's samples, found that all QC samples in the batch met the acceptance criteria and that there were no anomalies noted. However, a review of the double-blind audit samples submitted by the Internal Dosimetry Program (IDP) during the time frame in question, found three audit samples spiked at 10 dpm but with reported activity of less than 1 dpm. The three audit samples were likewise processed in a batch of nine samples around the same time frame as the worker's samples. At PNNL request, the analytical lab reviewed the likelihood that the three worker's samples from July 2009 and the three audit samples were switched and determined that there was reasonable evidence to suggest that the batches had been mislabeled and strontium results were incorrectly reported

Impact:

1. No impact on Hanford work assignments or dose control
2. Incorrect results were reported in the bioassay database for 18 workers.
3. A total of six special follow-up samples were unnecessarily collected and analyzed for the three workers.
4. Historically, it is theoretically possible that swapping samples could have led to follow-up of the wrong workers with the result that a low-level intake might have gone unreported

.

Cause:

The mix-up occurred when the sample planchettes for two separate batches were manually labeled with identical batch numbers. This resulted in confusion as to which group of planchettes was correctly labeled and which group was incorrectly labeled. Without consulting the Group Leader or Senior Analyst, the technician who labeled the planchettes determined incorrectly which group had the wrong batch labels.

Corrective Actions:

Action (1): Review recent strontium results in REX to identify other abnormal results.
Due - September 29, 2009 (completed September 23, 2009)

Owner - Cheryl Antonio
Documentation - A query of the recent strontium data in REX identified three samples that were submitted to the lab by the Internal Dosimetry Program (IDP) as double-blind QC spikes, but were reported as not exceeding the decision level. The samples were spiked with strontium at the 10 dpm contractual level – about the same level as the results for the three worker samples. The urine samples from the three workers with elevated strontium levels were processed about the same time as the double-blind QC spikes.

Action (2): Request the lab to review the three worker's samples and the three audit samples in light of a possible mix-up.
Due- September 29, 2009 (completed September 24, 2009)
Owner - JA MacLellan
Documentation – On September 24, 2009, the lab reported that there was reasonable evidence that the three worker samples and the three audit samples had been mixed up and that there was an error in reporting the correct sample results. The laboratory was instructed by the Battelle Technical Administrator to prepare and submit a formal incident report.

Action (3): Review past data for similar incidents
Due- September 30, 2009 (completed September 28)
Owner - . Jay Maclellan
Documentation –REX bioassay results reported by the current laboratory since contract was initiated in 2005 were reviewed. Out of approximately 50,000 results for 24,000 analyses, there were 318 results that exceeded the decision level or screening level were identified. Of those 318 there were 38 chosen for further review because they didn't appear to be supported by follow-up analyses. Of the 38 results only 6 were not explained by variable environmental contaminants or statistical variability. This evaluation didn't find any questionable results that hadn't been previously investigated. There was no evidence of switched samples with the six unresolved high results.

APPENDIX A

QUALITY CONTROL SAMPLE RESULTS
(Historical File Only)

Reprinted. Identified on 6/27/2012 that document not in error.

ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD 1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
AM241	1001	14	02/03/2010	10A0694	99159	IPA	79	0.0000	0.0000	F	J	-0.0125	0.0051	-	
AM241	1001	15	02/03/2010	10A0697	99204	IPA	230	0.0000	0.0000	F	J	0.0165	0.0095	-	
AM241	1001	18	02/03/2010	10A0690	YH402	IPA	144	0.0000	0.0000	F	J	-0.0027	0.0033	-	
AM241	1001	16	02/03/2010	10A0698	99204	IPA	102	0.0000	0.0000	F	J	-0.0064	0.0044	-	
AM241	1001	17	02/03/2010	10A0689	YH402	IPA	242	0.0000	0.0000	F	J	0.0464	0.0195	+	
				5	**F**	**AM241 Count**		**0.0000 5**	**Average Result St Dev**		**0.0083 0.0239**	**DL MDA**	**0.0510 0.1160**	**Chem Yield 0.86 Det Eff 0.39**	**Time 960**
AM241	1001	09	02/03/2010	10A0691	80098	IPA	171	1.5900	0.1590	F	J	1.7900	0.3670	+	0.1258
AM241	1001	12	02/03/2010	10A0696	99120	IPA	57	1.5900	0.1590	F	J	1.6600	0.3350	+	0.0440
AM241	1001	13	02/03/2010	10A0693	99159	IPA	75	1.5900	0.1590	F	J	1.4600	0.3010	+	-0.0818
AM241	1001	11	02/03/2010	10A0695	99120	IPA	78	1.5900	0.1590	F	J	1.7300	0.3510	+	0.0881
AM241	1001	10	02/03/2010	10A0692	80098	IPA	101	1.5900	0.1590	F	J	1.6000	0.3270	+	0.0063
				5	**F**	**AM241 Count**		**1.5900 5**	**Average Result St Dev**		**1.6480 0.1272**			**Mean Rel. Bias Mean Rel. Precision**	**0.0365 0.0800**
	Number of total F AM241 **10**														
PU238	1001	15	02/03/2010	10A0697	99204	IPA	230	0.0000	0.0000	F	J	-0.0014	0.0092	-	
PU238	1001	14	02/03/2010	10A0694	99159	IPA	79	0.0000	0.0000	F	J	-0.0052	0.0170	-	
PU238	1001	18	02/03/2010	10A0690	YH402	IPA	144	0.0000	0.0000	F	J	0.0084	0.0077	-	
PU238	1001	17	02/03/2010	10A0689	YH402	IPA	242	0.0000	0.0000	F	J	-0.0014	0.0071	-	
PU238	1001	16	02/03/2010	10A0698	99204	IPA	102	0.0000	0.0000	F	J	-0.0014	0.0059	-	
				5	**F**	**PU238 Count**		**0.0000 5**	**Average Result St Dev**		**-0.0002 0.0051**	**DL MDA**	**0.0108 0.0362**	**Chem Yield 0.83 Det Eff 0.39**	**Time 960**
PU238	1001	09	02/03/2010	10A0691	80098	IPA	171	0.8200	0.0820	F	J	1.0600	0.1380	+	0.2927
PU238	1001	11	02/03/2010	10A0695	99120	IPA	78	0.8200	0.0820	F	J	1.0100	0.1260	+	0.2317
PU238	1001	10	02/03/2010	10A0692	80098	IPA	101	0.8200	0.0820	F	J	0.6880	0.0948	+	-0.1610
PU238	1001	13	02/03/2010	10A0693	99159	IPA	75	0.8200	0.0820	F	J	0.7910	0.1080	+	-0.0354
PU238	1001	12	02/03/2010	10A0696	99120	IPA	57	0.8200	0.0820	F	J	0.8780	0.1090	+	0.0707
				5	**F**	**PU238 Count**		**0.8200 5**	**Average Result St Dev**		**0.8854 0.1533**			**Mean Rel. Bias Mean Rel. Precision**	**0.0798 0.1869**

Page 1 of 8

ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD	J	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
	Number of total F PU238 **10**															
PU239	1001	16	02/03/2010	10A0698		99204	IPA	102	0.0000	0.0000	F	J	-0.0059	0.0059	-	
PU239	1001	14	02/03/2010	10A0694		99159	IPA	79	0.0000	0.0000	F	J	-0.0059	0.0062	-	
PU239	1001	17	02/03/2010	10A0689		YH402	IPA	242	0.0000	0.0000	F	J	0.0059	0.0054	-	
PU239	1001	18	02/03/2010	10A0690		YH402	IPA	144	0.0000	0.0000	F	J	-0.0134	0.0298	-	
PU239	1001	15	02/03/2010	10A0697		99204	IPA	230	0.0000	0.0000	F	J	-0.0059	0.0092	-	
				5	**F**		**PU239 Count**		**0.0000 5**	**Average Result St Dev**		**-0.0050 0.0069**	**DL MDA**	**0.0148 0.0441**	**Chem Yield 0.83 Det Eff 0.39**	**Time 960**
PU239	1001	11	02/03/2010	10A0695		99120	IPA	78	1.0000	0.1000	F	J	1.0300	0.1270	+	0.0300
PU239	1001	09	02/03/2010	10A0691		80098	IPA	171	1.0000	0.1000	F	J	1.1300	0.1430	+	0.1300
PU239	1001	12	02/03/2010	10A0696		99120	IPA	57	1.0000	0.1000	F	J	0.9570	0.1150	+	-0.0430
PU239	1001	13	02/03/2010	10A0693		99159	IPA	75	1.0000	0.1000	F	J	0.9840	0.1250	+	-0.0160
PU239	1001	10	02/03/2010	10A0692		80098	IPA	101	1.0000	0.1000	F	J	0.7780	0.1030	+	-0.2220
				5	**F**		**PU239 Count**		**1.0000 5**	**Average Result St Dev**		**0.9758 0.1287**			**Mean Rel. Bias Mean Rel. Precision**	**-0.0242 0.1287**
	Number of total F PU239 **10**															
AM241	0904	07	04/22/2009	09D0454		3G544	IPSA	1429	0.0200	0.0004	U	L	0.0213	0.0070	+	0.0650
AM241	0904	08	04/22/2009	09D0446		99153	IPSA	1130	0.0200	0.0004	U	L	0.0260	0.0080	+	0.3000
AM241	0904	09	05/28/2009	09E0202		3C135	IPSA	1359	0.0200	0.0004	U	L	0.0200	0.0069	+	
AM241	0905	07	05/28/2009	09E0141		50575	IPSA	1155	0.0200	0.0005	U	L	0.0241	0.0074	+	0.2050
AM241	0905	06	05/28/2009	09E0076		91382	IPSA	1283	0.0200	0.0005	U	L	0.0176	0.0063	+	-0.1200
AM241	0905	08	06/26/2009	09F0432		99156	IPSA	1472	0.0200	0.0005	U	L	0.0147	0.0057	+	-0.2650
AM241	0908	08	08/26/2009	09H0443		51077	IPSA	1127	0.0200	0.0004	U	L	0.0190	0.0063	+	-0.0500
AM241	0908	07	08/26/2009	09H0180		99151	IPSA	1466	0.0200	0.0004	U	L	0.0225	0.0068	+	0.1250
AM241	0910	09	10/28/2009	09J0227		99161	IPSA	1412	0.0200	0.0004	U	L	0.0226	0.0068	+	0.1300
AM241	0910	10	11/27/2009	09K0145		59600	IPSA	1155	0.0200	0.0004	U	L	0.0217	0.0067	+	0.0850
AM241	1001	03	01/29/2010	10A0117		3C134	IPSA	1165	0.0200	0.0002	U	L	0.0187	0.0059	+	-0.0650
AM241	1001	04	01/29/2010	10A0118		3C142	IPSA	1225	0.0200	0.0002	U	L	0.0116	0.0046	+	-0.4200
AM241	1001	02	01/29/2010	10A0194		99158	IPSA	1394	0.0200	0.0002	U	L	0.0216	0.0067	+	0.0800
AM241	1001	05	02/10/2010	10A0749		91386	IPSA	1356	0.0200	0.0002	U	L	0.0200	0.0065	+	

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ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD 1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
AM241	1001	07	02/10/2010	10A0751	32533	IPSA	1275	0.0200	0.0002	U	L	0.0112	0.0045	+	-0.4400
AM241	1001	06	02/10/2010	10A0750	99150	IPSA	1103	0.0200	0.0002	U	L	0.0144	0.0050	+	-0.2800
AM241	1001	08	03/26/2010	10C0183	32476	IPSA	1407	0.0200	0.0002	U	L	0.0182	0.0059	+	-0.0900
AM241	1003	04	03/26/2010	10C0252	59001	IPSA	1384	0.0200	0.0004	U	L	0.0138	0.0050	+	-0.3100
				18	**U**	**AM241 Count**		**0.0200 18**	**Average Result St Dev**		**0.0188 0.0042**			**Mean Rel. Bias Mean Rel. Precision**	**-0.0583 0.2121**
AM241	0906	13	06/26/2009	09F0290	50809	IPSA	1367	0.1000	0.0004	U	L	0.1080	0.0246	+	0.0800
AM241	0906	14	06/26/2009	09F0431	59621	IPSA	1119	0.1000	0.0004	U	L	0.1470	0.0315	+	0.4700
AM241	0906	11	06/26/2009	09F0232	31776	IPSA	1226	0.1000	0.0004	U	L	0.0963	0.0219	+	-0.0370
AM241	0906	12	06/26/2009	09F0289	50807	IPSA	1149	0.1000	0.0004	U	L	0.0998	0.0221	+	-0.0020
AM241	0906	15	07/27/2009	09G0180	99159	IPSA	1266	0.1000	0.0004	U	L	0.1010	0.0230	+	0.0100
				5	**U**	**AM241 Count**		**0.1000 5**	**Average Result St Dev**		**0.1104 0.0209**			**Mean Rel. Bias Mean Rel. Precision**	**0.1042 0.2089**
	Number of total U AM241 **23**														
AM243	0904	02	04/16/2009	09D0298	91386	AM243	1331	0.0000	0.0000	U		-0.0021	0.0035	-	
AM243	0904	01	04/16/2009	09D0405	99120	AM243	1233	0.0000	0.0000	U		0.0011	0.0008	-	
AM243	0902	01	04/22/2009	09D0329	32533	AM243	1327	0.0000	0.0000	U		0.0005	0.0012	-	
AM243	0907	01	07/31/2009	09G0191	80098	AM243	1283	0.0000	0.0000	U		0.0031	0.0019	-	
AM243	0908	01	08/28/2009	09H0124	3C136	AM243	1309	0.0000	0.0000	U		0.0032	0.0020	-	
AM243	0908	02	09/24/2009	09I0427	99152	AM243	1304	0.0000	0.0000	U		-0.0004	0.0026	-	
				6	**U**	**AM243 Count**		**0.0000 6**	**Average Result St Dev**		**0.0009 0.0021**	**DL MDA**	**0.0042 0.0131**	**Chem Yield 0.87 Det Eff 0.39**	**Time 2520**
	Number of total U AM243 **6**														
PU238	0904	08	04/21/2009	09D0446	99153	IPSA	1130	0.0000	0.0000	U	L	0.0023	0.0023	-	
PU238	0904	07	04/21/2009	09D0454	3G544	IPSA	1429	0.0000	0.0000	U	L	0.0012	0.0020	-	
PU238	0904	09	05/28/2009	09E0202	3C135	IPSA	1359	0.0000	0.0000	U	L	0.0036	0.0027	-	
PU238	0905	06	05/28/2009	09E0076	91382	IPSA	1283	0.0000	0.0000	U	L	-0.0004	0.0033	-	
PU238	0905	07	05/28/2009	09E0141	50575	IPSA	1155	0.0000	0.0000	U	L	0.0009	0.0022	-	
PU238	0906	14	06/26/2009	09F0431	59621	IPSA	1119	0.0000	0.0000	U	L	0.0023	0.0028	-	
PU238	0906	12	06/26/2009	09F0289	50807	IPSA	1149	0.0000	0.0000	U	L	-0.0011	0.0023	-	

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ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD 1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
PU238	0906	13	06/26/2009	09F0290	50809	IPSA	1367	0.0000	0.0000	U	L	0.0014	0.0026	-	
PU238	0906	11	06/26/2009	09F0232	31776	IPSA	1226	0.0000	0.0000	U	L	-0.0014	0.0023	-	
PU238	0905	08	06/26/2009	09F0432	99156	IPSA	1472	0.0000	0.0000	U	L	0.0025	0.0025	-	
PU238	0906	15	07/27/2009	09G0180	99159	IPSA	1266	0.0000	0.0000	U	L	-0.0002	0.0031	-	
PU238	0907	05	07/30/2009	09G0192	80098	IPU	1283	0.0000	0.0000	U	Q	0.0009	0.0018	-	
PU238	0908	08	08/24/2009	09H0443	51077	IPSA	1127	0.0000	0.0000	U	L	0.0000	0.0024	-	
PU238	0908	07	08/24/2009	09H0180	99151	IPSA	1466	0.0000	0.0000	U	L	0.0047	0.0033	-	
PU238	0907	07	08/24/2009	09H0120	99162	IPU	1408	0.0000	0.0000	U	L	0.0011	0.0024	-	
PU238	0910	09	10/28/2009	09J0227	99161	IPSA	1412	0.0000	0.0000	U	L	-0.0013	0.0020	-	
PU238	0910	10	12/04/2009	09K0145	59600	IPSA	1155	0.0000	0.0000	U	L	0.0040	0.0030	-	
PU238	1001	02	01/29/2010	10A0194	99158	IPSA	1394	0.0000	0.0000	U	L	0.0042	0.0030	-	
PU238	1001	04	01/29/2010	10A0118	3C142	IPSA	1225	0.0000	0.0000	U	L	0.0031	0.0023	-	
PU238	1001	03	01/29/2010	10A0117	3C134	IPSA	1165	0.0000	0.0000	U	L	0.0019	0.0019	-	
PU238	1001	07	02/08/2010	10A0751	32533	IPSA	1275	0.0000	0.0000	U	L	0.0000	0.0023	-	
PU238	1001	05	02/08/2010	10A0749	91386	IPSA	1356	0.0000	0.0000	U	L	-0.0014	0.0025	-	
PU238	1001	06	02/08/2010	10A0750	99150	IPSA	1103	0.0000	0.0000	U	L	0.0053	0.0032	-	
PU238	1003	04	03/26/2010	10C0252	59001	IPSA	1384	0.0000	0.0000	U	L	-0.0003	0.0023	-	
PU238	1001	08	03/26/2010	10C0183	32476	IPSA	1407	0.0000	0.0000	U	L	-0.0014	0.0021	-	
					25	**U**	**PU238 Count**	**0.0000**	**Average Result**		**0.0013**	**DL**	**0.0035**	**Chem Yield**	**0.74**	**Time**	**2520**
								25	**St Dev**		**0.0021**	**MDA**	**0.0111**	**Det Eff**	**0.39**		
	Number of total U PU238 **25**																
PU239	0904	08	04/21/2009	09D0446	99153	IPSA	1130	0.0200	0.0005	U	L	0.0233	0.0059	+	0.1650
PU239	0904	07	04/21/2009	09D0454	3G544	IPSA	1429	0.0200	0.0005	U	L	0.0197	0.0052	+	-0.0150
PU239	0905	07	05/28/2009	09E0141	50575	IPSA	1155	0.0200	0.0005	U	L	0.0200	0.0057	+	
PU239	0904	09	05/28/2009	09E0202	3C135	IPSA	1359	0.0200	0.0005	U	L	0.0210	0.0063	+	0.0500
PU239	0905	06	05/28/2009	09E0076	91382	IPSA	1283	0.0200	0.0005	U	L	0.0262	0.0066	+	0.3100
PU239	0905	08	06/26/2009	09F0432	99156	IPSA	1472	0.0200	0.0005	U	L	0.0205	0.0058	+	0.0250
PU239	0907	05	07/30/2009	09G0192	80098	IPU	1283	0.0200	0.0006	U	Q	0.0174	0.0050	+	-0.1300
PU239	0908	07	08/24/2009	09H0180	99151	IPSA	1466	0.0200	0.0005	U	L	0.0226	0.0054	+	0.1300
PU239	0908	08	08/24/2009	09H0443	51077	IPSA	1127	0.0200	0.0005	U	L	0.0191	0.0053	+	-0.0450

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ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD_1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
PU239	0907	07	08/24/2009	09H0120	99162	IPU	1408	0.0200	0.0006	U	L	0.0267	0.0058	+	0.3350
PU239	0910	09	10/28/2009	09J0227	99161	IPSA	1412	0.0200	0.0005	U	L	0.0292	0.0064	+	0.4600
PU239	0910	10	12/04/2009	09K0145	59600	IPSA	1155	0.0200	0.0005	U	L	0.0201	0.0059	+	0.0050
PU239	1001	04	01/29/2010	10A0118	3C142	IPSA	1225	0.0200	0.0003	U	L	0.0295	0.0064	+	0.4750
PU239	1001	03	01/29/2010	10A0117	3C134	IPSA	1165	0.0200	0.0002	U	L	0.0219	0.0059	+	0.0950
PU239	1001	02	01/29/2010	10A0194	99158	IPSA	1394	0.0200	0.0003	U	L	0.0148	0.0048	+	-0.2600
PU239	1001	06	02/08/2010	10A0750	99150	IPSA	1103	0.0200	0.0003	U	L	0.0250	0.0062	+	0.2500
PU239	1001	05	02/08/2010	10A0749	91386	IPSA	1356	0.0200	0.0003	U	L	0.0174	0.0066	+	-0.1300
PU239	1001	07	02/08/2010	10A0751	32533	IPSA	1275	0.0200	0.0003	U	L	0.0204	0.0057	+	0.0200
PU239	1001	08	03/26/2010	10C0183	32476	IPSA	1407	0.0200	0.0003	U	L	0.0243	0.0061	+	0.2150
PU239	1003	04	03/26/2010	10C0252	59001	IPSA	1384	0.0200	0.0005	U	L	0.0175	0.0046	+	-0.1250
				20	**U**	**PU239 Count**		**0.0200 20**	**Average Result St Dev**			**0.0218 0.0040**			**Mean Rel. Bias 0.0915 Mean Rel. Precision 0.2004**
PU239	0906	13	06/26/2009	09F0290	50809	IPSA	1367	0.1000	0.0005	U	L	0.1030	0.0138	+	0.0300
PU239	0906	12	06/26/2009	09F0289	50807	IPSA	1149	0.1000	0.0005	U	L	0.1080	0.0125	+	0.0800
PU239	0906	14	06/26/2009	09F0431	59621	IPSA	1119	0.1000	0.0005	U	L	0.1040	0.0126	+	0.0400
PU239	0906	11	06/26/2009	09F0232	31776	IPSA	1226	0.1000	0.0005	U	L	0.0951	0.0126	+	-0.0490
PU239	0906	15	07/27/2009	09G0180	99159	IPSA	1266	0.1000	0.0005	U	L	0.0933	0.0124	+	-0.0670
				5	**U**	**PU239 Count**		**0.1000 5**	**Average Result St Dev**			**0.1007 0.0062**			**Mean Rel. Bias 0.0068 Mean Rel. Precision 0.0624**
	Number of total U PU239 **25**														
SR	0904	07	04/24/2009	09D0454	3G544	IPSA	1429	10.0000	0.1200	U	L	10.3000	1.3600	+	0.0300
SR	0904	08	04/24/2009	09D0446	99153	IPSA	1130	10.0000	0.1200	U	L	10.6000	1.3700	+	0.0600
SR	0905	07	05/28/2009	09E0141	50575	IPSA	1155	10.0000	0.1370	U	L	8.8800	1.2200	+	-0.1120
SR	0904	09	05/28/2009	09E0202	3C135	IPSA	1359	10.0000	0.1370	U	L	9.5300	1.2900	+	-0.0470
SR	0905	06	05/28/2009	09E0076	91382	IPSA	1283	10.0000	0.1370	U	L	11.7000	1.5300	+	0.1700
SR	0906	12	06/24/2009	09F0289	50807	IPSA	1149	10.0000	0.1500	U	L	8.5800	1.3100	+	-0.1420
SR	0906	14	06/24/2009	09F0431	59621	IPSA	1119	10.0000	0.1500	U	L	9.5100	1.4400	+	-0.0490
SR	0906	13	06/24/2009	09F0290	50809	IPSA	1367	10.0000	0.1500	U	L	8.1200	1.4100	+	-0.1880
SR	0906	11	06/24/2009	09F0232	31776	IPSA	1226	10.0000	0.1500	U	L	9.6600	1.5000	+	-0.0340

ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD 1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
SR	0905	08	06/24/2009	09F0432	99156	IPSA	1472	10.0000	0.1370	U	L	11.1000	1.8500	+	0.1100
SR	0906	15	07/30/2009	09G0180	99159	IPSA	1266	10.0000	0.1500	U	L	7.6600	1.0200	+	-0.2340
SR	0908	07	08/27/2009	09H0180	99151	IPSA	1466	10.0000	0.1850	U	L	8.7900	1.1500	+	-0.1210
SR	0908	08	08/27/2009	09H0443	51077	IPSA	1127	10.0000	0.1850	U	L	9.5400	1.2300	+	-0.0460
SR	0910	09	11/02/2009	09J0227	99161	IPSA	1412	10.0000	0.1790	U	L	11.6000	1.4200	+	0.1600
SR	0910	10	11/25/2009	09K0145	59600	IPSA	1155	10.0000	0.1790	U	L	10.6000	1.3800	+	0.0600
SR	1001	03	01/26/2010	10A0117	3C134	IPSA	1165	10.0000	0.1100	U	L	8.9700	1.2000	+	-0.1030
SR	1001	04	01/26/2010	10A0118	3C142	IPSA	1225	10.0000	0.1100	U	L	9.1900	1.1400	+	-0.0810
SR	1001	02	01/27/2010	10A0194	99158	IPSA	1394	10.0000	0.1100	U	L	9.2900	1.1500	+	-0.0710
SR	1001	07	02/04/2010	10A0751	32533	IPSA	1275	10.0000	0.1100	U	L	8.5000	1.1300	+	-0.1500
SR	1001	05	02/05/2010	10A0749	91386	IPSA	1356	10.0000	0.1100	U	L	7.5400	1.0300	+	-0.2460
SR	1001	06	02/05/2010	10A0750	99150	IPSA	1103	10.0000	0.1100	U	L	8.1000	1.0900	+	-0.1900
SR	1001	08	03/26/2010	10C0183	32476	IPSA	1407	10.0000	0.1100	U	L	11.2000	1.4700	+	0.1200
SR	1003	04	03/26/2010	10C0252	59001	IPSA	1384	10.0000	0.1790	U	L	10.6000	1.3800	+	0.0600
				23	**U**	**SR Count**		**10.0000**	**Average Result**			**9.5461**			**Mean Rel. Bias** **-0.0454**
								23	**St Dev**			**1.2284**			**Mean Rel. Precision** **0.1228**

Number of total U SR **23**

U 235	0906	07	08/15/2009	09F1225	AU001	IU	977	0.0000	0.0000	U	U	0.0063	0.0039	-	
U 235	0906	09	08/15/2009	09F1227	AU001	IU	979	0.0000	0.0000	U	U	0.0074	0.0053	-	
U 235	0906	04	08/15/2009	09F1222	AU001	IU	978	0.0000	0.0000	U	U	0.0023	0.0028	-	
U 235	0906	06	08/15/2009	09F1224	AU001	IU	979	0.0000	0.0000	U	U	0.0026	0.0032	-	
U 235	0906	10	08/15/2009	09F1228	AU001	IU	978	0.0000	0.0000	U	U	0.0014	0.0038	-	
U 235	0906	08	08/15/2009	09F1226	AU001	IU	978	0.0000	0.0000	U	U	0.0044	0.0039	-	
U 235	0906	05	08/15/2009	09F1223	AU001	IU	977	0.0000	0.0000	U	U	0.0078	0.0042	-	
U 235	0910	06	02/02/2010	10A0833	AU001	IU	1285	0.0000	0.0000	U	U	0.0058	0.0036	-	
U 235	0911	04	02/02/2010	10A0832	AU001	IU	1243	0.0000	0.0000	U	U	0.0057	0.0040	-	
U 235	0911	05	02/02/2010	10A0831	AU001	IU	1282	0.0000	0.0000	U	U	0.0059	0.0037	-	
				10	**U**	**U 235 Count**		**0.0000**	**Average Result**			**0.0050**	**DL**	**0.0040**	**Chem Yield** **0.87** **Time** **2520**
								10	**St Dev**			**0.0022**	**MDA**	**0.0120**	**Det Eff** **0.39**

Number of total U U 235 **10**

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ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
U 238	0906	05	08/15/2009	09F1223	AU001	IU	977	0.1500	0.0015	U	U	0.1760	0.0197	+	0.1733
U 238	0906	07	08/15/2009	09F1225	AU001	IU	977	0.1500	0.0015	U	U	0.1540	0.0179	+	0.0267
U 238	0906	06	08/15/2009	09F1224	AU001	IU	979	0.1500	0.0015	U	U	0.1270	0.0146	+	-0.1533
U 238	0906	08	08/15/2009	09F1226	AU001	IU	978	0.1500	0.0015	U	U	0.1550	0.0174	+	0.0333
U 238	0906	10	08/15/2009	09F1228	AU001	IU	978	0.1500	0.0015	U	U	0.1500	0.0168	+	
U 238	0906	04	08/15/2009	09F1222	AU001	IU	978	0.1500	0.0015	U	U	0.1380	0.0145	+	-0.0800
U 238	0906	09	08/15/2009	09F1227	AU001	IU	979	0.1500	0.0015	U	U	0.1550	0.0178	+	0.0333
U 238	0910	06	02/02/2010	10A0833	AU001	IU	1285	0.1500	0.0011	U	U	0.1490	0.0179	+	-0.0067
U 238	0911	05	02/02/2010	10A0831	AU001	IU	1282	0.1500	0.0024	U	U	0.1620	0.0188	+	0.0800
U 238	0911	04	02/02/2010	10A0832	AU001	IU	1243	0.1500	0.0024	U	U	0.1610	0.0184	+	0.0733
				10	**U**	**U 238 Count**		**0.1500 10**	**Average Result St Dev**		**0.1527 0.0134**				**Mean Rel. Bias 0.0180 Mean Rel. Precision 0.0891**
U 238	0905	03	05/15/2009	09E0227	59783	U 238	1404	0.2000	0.0032	U		0.1670	0.0147	+	-0.1650
U 238	0905	02	05/15/2009	09E0203	3C136	U 238	1144	0.2000	0.0032	U		0.2020	0.0135	+	0.0100
U 238	0905	05	06/16/2009	09F0364	80109	U 238	0000	0.2000	0.0032	U		0.1680	0.0139	+	-0.1600
U 238	0907	02	07/20/2009	09G0097	32514	U 238	1144	0.2000	0.0019	U		0.1780	0.0211	+	-0.1100
U 238	0907	03	07/23/2009	09G0126	32533	U 238	1398	0.2000	0.0019	U		0.1920	0.0082	+	-0.0400
U 238	0908	05	08/14/2009	09H0142	59600	U 238	1228	0.2000	0.0016	U		0.1920	0.0145	+	-0.0400
U 238	0908	03	08/14/2009	09H0442	32472	U 238	1141	0.2000	0.0016	U		0.1910	0.0136	+	-0.0450
U 238	0908	04	08/14/2009	09H0125	3C142	U 238	1398	0.2000	0.0016	U		0.1890	0.0063	+	-0.0550
U 238	0907	04	08/14/2009	09H0119	99161	U 238	1228	0.2000	0.0019	U		0.1810	0.0068	+	-0.0950
U 238	0908	06	09/19/2009	09I0291	91386	U 238	1276	0.2000	0.0016	U		0.1900	0.0076	+	-0.0500
U 238	0910	07	10/14/2009	09J0238	80076	U 238	1095	0.2000	0.0011	U		0.1410	0.0174	+	-0.2950
U 238	0910	03	10/14/2009	09J0149	50784	U 238	1138	0.2000	0.0011	U		0.1820	0.0058	+	-0.0900
U 238	0910	08	10/14/2009	09J0289	3G522	U 238	1266	0.2000	0.0011	U		0.1960	0.0063	+	-0.0200
U 238	0910	04	11/12/2009	09K0131	3C136	U 238	1396	0.2000	0.0011	U		0.1680	0.0096	+	-0.1600
U 238	0911	03	11/12/2009	09K0146	59783	U 238	1396	0.2000	0.0024	U		0.1600	0.0073	+	-0.2000
U 238	0911	02	11/19/2009	09K0301	SG563	U 238	1137	0.2000	0.0024	U		0.2040	0.0062	+	0.0200
U 238	1003	02	03/23/2010	10C0311	99156	U 238	1135	0.2000	0.0012	U		0.4330	0.0208	+	1.1650

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ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD_1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
				17	U	U 238		0.2000	Average Result		0.1961			Mean Rel. Bias	-0.0194
						Count		17	St Dev		0.0632			Mean Rel. Precision	0.3159

Number of total U U 238 27

Total Samples	68
Total Results	169

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APPENDIX B

GEL QUALITY CONTROL SAMPLE REPORT SUMMARY
(Historical File Only)

PNNL
QUARTERLY
QC PACKAGE

Annual for 2009
April 1, 2009 – March 31, 2010

Data was reviewed and found acceptable.

Reviewed By: Date: 5-26-10

Table of Contents

Tritium – Blank Activity
Tritium – LCS Bias Low
U-232 – Tracer Yield
U-233/234 – Blank Activity
U-235/236 – Blank Activity
U-238 – Blank Activity
U-238 – LCS Bias High
U-238 – LCS Bias Low
U-236 – Blank Activity
U-236 – Duplicate
U-236 – Matrix Spike
U-236 – LCS Bias High/Low
U-233 – Tracer Yield
U-238 – ICP-MS – Blank Activity
U-238 – ICP-MS – Duplicate
U-238 – ICP-MS – Matrix Spike
U-238 – ICP-MS – LCS Bias High
U-238 – ICP-MS – LCS Bias Low

Fecal Data
Am-241 – Blank Activity
Am-241 – Duplicate RER
Am-241 – LCS Bias High
Am-241 – LCS Bias Low
Am-243 – Tracer Yield
Pu-238 – Blank Activity
Pu-238 – Duplicate RER
Pu-239/240 – Blank Activity
Pu-239/240 – Duplicate RER
Pu-239/240 – LCS Bias High
Pu-239/240 – LCS Bias Low
Pu-242 – Tracer Yield

Section 3: NRIP Results (Only for Annual Package)
NRIP Fecal
NRIP Urine

Legend

#	= the N-value (number of the samples in the data set)
Samp ID	= GEL laboratory sample identification number
Inst	= the analytical instrument identification number/name
Run Date	= the sample analysis date
LCL	= Lower Control Level (minus 3 sigma)
LWL	= Lower Warning Level (minus 2 sigma)
Mean	= the average value of the data set
Numvalue	= Number Value for parameter being monitored
Exclude	= a checked box indicates the data was not used in the calculation of the mean and control limits
Stdev	= Standard Deviation
UWL	= Upper Warning Level (plus 2 sigma)
UCL	= Upper Contol Level (plus 3 sigma)
Dispersion	= the difference of the individual relative bias from the mean
Parent Sample	= the sample that was duplicated
TPU	= Total Proportion Uncertainty (1 sigma combined standard uncertainty)
RER	= Relative Error Ratio (the difference of the individual duplicate pairs based on the combined standard uncertainties of the individual analyses)
Nominal	= the calculated concentration of the spike in the sample geometry
Result	= the actual measured analyte concentration in the sample
Bias	= the deviation of a measured value from the expected value

Statistical Parameters Utilized by The GEL Group, Inc

Zone Definitions

Zone A – Area defined as being between 2 and 3 times sigma above the center line
Zone B – Area defined as being between 1 and 2 times sigma above the center line
Zone C – Area defined as being between the center line and 1 times sigma

Data Flag Definitions

1. Nine (9) points on Zone C and beyond on one side of the central line - Indicates that the process average may have changed
2. Six (6) points in a row steadily increasing or decreasing on one side of the central line – Indicates that a drift may be occurring in the process average
3. Fourteen (14) points in a row alternating up or down on either side of the center line – If this test is positive it indicates that two systematically alternating causes may be producing different results
4. Two (2) out of three (3) points in a row are in Zone A or beyond – Indicates an early warning of a process shift
5. Four (4) out of five (5) points are in Zone B or beyond – If positive, this, like flag 4, indicates and early warning of a potential process shift
6. Fifteen (15) points are in Zone C above or below the center line – Indicates a smaller variability than expected
7. Eight (8) points in a row are in Zone B, A or beyond on either side of the center line with no points occurring in Zone C – Indicates that different samples are affected by different factors resulting in bimodal distribution of averages

References

Statistica Software – Data Mining, Statistical Analysis and Quality Control
Quality Control Charts – www.statsoft.com/textbook/stquacon.html

SECTION 1

CASE NARRATIVE

Annual - QC Report Operational Year 2009

This report summarizes Quality Control Samples (QC) analyzed with bioassay samples under Contract 11530 effective date ending 2/23/10 and Contract 112512 effective date beginning 2/24/10. Included in the report are listings for the blank, duplicate and spike results. A description of the attached data is provided below. Twelve thousand four hundred and fifty-two reported samples were analyzed under this contract with a run date during the contract year including failed analyses, recounts, and reanalyses.

PNNL Sample/QC Summary

Test Description	Matrix	Reported Samples	QC Samples	Total Samples	% QC
Americium	Fecal	73	88	161	55
Plutonium	Fecal	72	88	160	55
Neptunium	Urine	13	24	37	65
Americium-243	Urine	109	108	217	50
Thorium	Urine	25	48	73	66
ICP-MS Total Uranium (U-238)	Urine	1399	709	2108	34
ICP-MS Uranium-236	Urine	20	17	37	46
Americium	Urine	1407	701	2108	33
Plutonium	Urine	4097	1742	5839	30
Strontium 90	Urine	1465	687	2152	32
Total Strontium	Urine	216	195	411	47
Tritium	Urine	1289	388	1677	23
Uranium	Urine	791	465	1256	37
Totals		12452	6022	18474	33

Blanks

The following table contains the analyses, isotope, matrix, and the calculated MDAs. The alpha spectrometry MDAs are based on the average blank counts and average tracer yields for the year. The Strontium MDAs are adjusted according to the average tracer yield for the year. The Uranium by ICP-MS MDAs are based on the standard deviation of a standard analyzed each day when samples are analyzed throughout the year.

PNNL
Annual for 2009

Isotope	Matrix	N#	MDA	Lc	Avg. Volume	Sample units	Detector Yield	Count Efficiency	Time (min)
Am-241	Urine	232	0.010	0.00415	1	dpm/s	0.8666	0.391	2520
Am-243	Urine	36	0.013	0.00555	1	dpm/s	0.9221	0.391	2520
Cm-242	Urine	84	0.010	0.00389	1	dpm/s	0.8666	0.391	2520
Cm-243/244	Urine	232	0.011	0.00431	1	dpm/s	0.8666	0.391	2520
Np-237	Urine	8	0.006	0.01103	1	dpm/s	0.7169	0.391	2520
Pu-238	Urine	570	0.010	0.00404	1	dpm/s	0.9017	0.391	2520
Pu-239/240	Urine	570	0.011	0.00441	1	dpm/s	0.9017	0.391	2520
Th-228	Urine	16	0.040	0.02630	1	dpm/s	0.899	0.386	2520
Th-229	Urine	15	0.015	0.00776	1	dpm/s	0.899	0.386	2520
Th-230	Urine	16	0.031	0.01810	1	dpm/s	0.899	0.386	2520
Th-232	Urine	16	0.015	0.00703	1	dpm/s	0.899	0.386	2520
U-233/234	Urine	149	0.021	0.00892	1	dpm/s	0.8622	0.386	2520
U-235/236	Urine	149	0.013	0.00551	1	dpm/s	0.8622	0.386	2520
U-238	Urine	149	0.020	0.00841	1	dpm/s	0.8622	0.386	2520
Sr-90	Urine	226	4.370	0.70143	1	dpm/s	0.759	0.379	45
Total Sr	Urine	65	4.504	0.82466	1	dpm/s	0.778	0.379	45
Tritium	Urine	191	0.826	0.53710	0.01 L	dpm/L	n/a	0.243	20
U-236 (ICPMS)	Urine	5	26.480	26.4800	0.5	pg/s	0.847	n/a	n/a
U-238 (ICPMS)	Urine	151	0.014	0.0141	0.001L	ug/s	n/a	n/a	n/a
Am-241	Fecal	22	0.048	0.00695	0.3333	dpm/s	0.865	0.391	960
Pu-238	Fecal	22	0.050	0.00787	0.3333	dpm/s	0.801	0.391	960
Pu-239/240	Fecal	22	0.054	0.00971	0.3333	dpm/s	0.801	0.391	960

*U-238 ICPMS MDA uses a 1:15 dilution factor

All analytical batches were analyzed with either a reagent blank, matrix blank or both. Blanks are in control when the calculated MDA and blank activity are both less than CRDL (contract required detection limit). In addition, the chemical tracer yields are evaluated against the yield requirements stated in the subject contract. For U-238 (ICP-MS) analysis and Tritium analysis, a yield monitor is not available and minimal chemistry is performed. Therefore a yield monitor is not used, and the yield is assumed to be 1 (100%). Overall, the blank data for each analytical process demonstrate the analyses were in control. Processing categories and samples which did not meet contractual requirements are discussed in the **Observations** section of this report.

Laboratory Control Samples (LCS)

The enclosed listing contains the analysis isotope, matrix, average relative bias and the relative precision statistic. One or more LCS sample was analyzed with each batch of samples

Test	Matrix	Number In Set (N#)	Range High	Average Nominal (dpm/sample)*	Average Relative Bias	Relative Precision
Americium-241	FECAL	19	High	5.785	-0.0146	0.0943
Plutonium-239/240	FECAL	19	High	5.819	-0.0211	0.0528
Uranium-238 Mass	URINE	183	High	0.888 ug/sample	0.0016	0.0794
Uranium-236 Mass	URINE	4	High	1280.91 pg/sample	-0.0183	0.0186
Americium-241	URINE	232	High	0.571	-0.0212	0.0985
Americium-243	URINE	36	High	0.495	0.0225	0.088
Curium-243/244	URINE	81	High	4.56	0.0143	0.0872
Neptunium-237	URINE	8	High	1.87	0.0901	0.0539
Plutonium-239/240	URINE	569	High	0.436	-0.0278	0.0803
Thorium-232	URINE	16	High	2.15	0.0403	0.0737
Uranium-238	URINE	147	High	0.373	-0.0252	0.107
Strontium-90	URINE	225	High	43.1	0.013	0.0871
Total Strontium	URINE	65	High	42.9	0.0259	0.0843

*Unless otherwise noted.

Test	Matrix	Number In Set (N#)	Range Low	Average Nominal (dpm/sample)*	Number Below Lc	Average Relative Bias	Relative Precision
Americium-241	FECAL	19	Low	0.575	0	0.1839	0.2333
Plutonium-239/240	FECAL	19	Low	0.242	0	0.0661	0.2468
Uranium-238 Mass	URINE	102	Low	0.0505 ug/sample	0	0.061	0.1224
Uranium-236 Mass	URINE	4	Low	128.07 pg/sample	0	-0.0005	0.0225
Americium-241	URINE	231	Low	0.0212	0	0.0681	0.289
Americium-243	URINE	36	Low	0.0204	0	-0.0471	0.2347
Curium-243/244	URINE	80	Low	0.0235	0	0.1226	0.2755
Neptunium-237	URINE	8	Low	0.0217	0	0.1616	0.2125
Plutonium-239/240	URINE	569	Low	0.0217	1	0.00534	0.249
Thorium-232	URINE	16	Low	0.109	0	0.017	0.102
Uranium-238	URINE	147	Low	0.0228	0	0.1072	0.4123
Strontium-90	URINE	225	Low	10.0	0	0.0369	0.143
Total Strontium	URINE	65	Low	10.384	0	0.0167	0.1319
Tritium	URINE	191	Low	5.79 pCi/mL	0	0.0213	0.0759

Overall, the LCS data demonstrates the analytical processes were in control. Any LCS outside the limits is discussed in the **Observations** section of this report.

Duplicate Samples (DUP)

The duplicate samples were evaluated to determine that the aliquot procedure produces results within the RER limits of 0 to 3.

Americium-241										
#	Sample ID	Inst	Run Date	Tracer Yield	Mean	RER	TPU	Parent Sample	Result	TPU
1	1201828766	1657	07-MAY-09	0.911	1.41	0.288	0.00865	228835001	0.013 and 0.0168	0.00865 and 0.00996
2	1201823114	1648	01-MAY-09	0.971	1.41	2.5	0.00454	228306002	0.00645 and -0.0108	0.00454 and 0.0052
3	1201831004	1671	12-MAY-09	1.03	1.41	1.74	0.00362	228983001	-0.00362 and 0.00725	0.00362 and 0.0051
4	1201880376	1660	17-JUL-09	0.911	1.41	1.29	0.0024	232921002	-0.00869 and -0.0269	0.0024 and 0.0139
5	1201888016	1683	31-JUL-09	0.786	1.41	.838	0.0486	234008001	0.149 and 0.0982	0.0486 and 0.0362
6	1201925009	1631	22-SEP-09	0.87	1.41	1.93	0.00547	236858001	-0.00908 and 0.0074	0.00547 and 0.00655
7	1201957825	1635	06-NOV-09	0.822	1.41	2.41	0.0309	239887001	0.0807 and 0.00566	0.0309 and 0.00399
8	1202014406	1648	22-JAN-10	0.807	1.41	1.43	0.00579	244560001	-0.0139 and -0.00457	0.00579 and .00302
9	1202015399	1675	03-FEB-10	0.822	1.41	.711	0.0159	244666001	0.0285 and 0.0464	0.0159 and 0.0195
10	1202044774	1643	24-FEB-10	0.91	1.41	1.7	0.00202	247082001	-0.00169 and 0.00796	0.00202 and 0.00532
11	1202045684	1666	24-FEB-10	0.781	1.41	.187	0.00281	247251001	-0.00166 and -0.00101	0.00281 and 0.00204

12	1202048182	1639	27-FEB-10	0.887	1.41	1.38	0.00271	247392001	-0.00853 and -0.00343	0.00271 and 0.00251
13	1202065391	1635	17-MAR-10	0.928	1.41	1.68	0.00265	248896001	-0.00816 and -0.00203	0.00265 and 0.00251
14	1202069652	1701	18-MAR-10	0.839	1.41	2.27	0.00271	249227001	-0.000645 and -0.0148	0.00271 and 0.00563
15	1202075428	1725	24-MAR-10	0.824	1.41	0.835	0.00304	249667001	-0.00526 and -0.0156	0.00304 and 0.012

Plutonium-238										
#	Sample ID	Inst	Run Date	Tracer Yield	Mean	RER	TPU	Parent Sample	Result	TPU
1	1201828770	1642	08-MAY-09	0.531	0.524	0.507	0.0103	228835001	0.00837 and -0.0091	0.0103 and 0.0329
2	1201831011	1676	12-MAY-09	0.447	0.524	0	0.0105	228983001	-0.000805 and -0.000805	0.0105 and 0.0099
3	1201880380	1666	17-JUL-09	0.856	0.524	0.116	0.015	232921002	-0.00532 and -0.00879	0.015 and 0.0258
4	1201888030	1688	31-JUL-09	0.739	0.524	0.413	0.0124	234008001	0.0153 and 0.0237	0.0124 and 0.0161
5	1201925017	1670	22-SEP-09	0.557	0.524	0.853	0.00807	236858001	0.00659 and -0.00766	0.00807 and 0.022
6	1201957829	1640	06-NOV-09	0.776	0.524	0.75	0.0166	239887001	-0.00509 and 0.0087	0.0166 and 0.00789
7	1202010115	1723	14-JAN-10	0.607	0.524	0.22	0.0212	243991001	-0.00521 and -0.000397	0.0212 and 0.00555
8	1202014423	1654	22-JAN-10	0.793	0.524	0	0.00576	244560001	-0.000757 and -0.000757	0.00576 and 0.00541

9	1202015403	1661	03-FEB-10	0.582	0.524	1.02	0.0102	244666001	0.0113 and -0.00136	0.0102 and 0.00708
10	1202044778	1656	24-FEB-10	0.96	0.524	0.87	0.014	247082001	0.0282 and 0.0136	0.014 and 0.00925
11	1202045689	1670	24-FEB-10	0.777	0.524	0.465	0.0161	247251001	0.0285 and 0.0189	0.0161 and 0.0129
12	1202048186	1648	27-FEB-10	0.76	0.524	0.649	0.00819	247392001	0.00903 and -0.00368	0.00819 and 0.0178
13	1202065395	1672	17-MAR-10	0.936	0.524	0.308	0.00522	248896001	0.00207 and 0	0.00522 and 0.00425
14	1202069656	1706	18-MAR-10	0.845	0.524	0.963	0.0108	249227001	0.0159 and 0.00432	0.0108 and 0.00529
15	1202075432	1717	24-MAR-10	0.852	0.524	0.72	0.00617	249667001	0.00681 and -0.00493	0.00617 and 0.0151

Plutonium-239/240										
#	Sample ID	Inst	Run Date	Tracer Yield	Mean	RER	TPU	Parent Sample	Result	TPU
1	1202010115	1723	14-JAN-10	0.607	0.878	0.233	0.00545	243991001	0.00358 and 0.00527	0.00545 and 0.00478
2	1202014423	1654	22-JAN-10	0.793	0.878	1.11	0.028	244560001	-0.0115 and 0.0235	0.028 and 0.0143
3	1202015403	1661	03-FEB-10	0.582	0.878	1.27	0.00758	244666001	-0.00591 and 0.00591	0.00758 and 0.00536
4	1202044778	1656	24-FEB-10	0.96	0.878	1.17	0.00578	247082001	0.0085 and -0.00805	0.00578 and 0.0129

5	1202045689	1670	24-FEB-10	0.777	0.878	0.798	0.00425	247251001	0.000204 and - 0.00574	0.00425 and 0.00612
6	1202048186	1648	27-FEB-10	0.76	0.878	0.694	0.00422	247392001	0.00465 and - 0.00805	0.00422 and 0.0178
7	1202065395	1672	17-MAR-10	0.936	0.878	1.32	0.00501	248896001	0.00553 and 0.0239	0.00501 and 0.013
8	1202069656	1706	18-MAR-10	0.845	0.878	1.28	0.00751	249227001	0.00751 and 0.0295	0.00751 and 0.0155
9	1202075432	1717	24-MAR-10	0.852	0.878	0.545	0.00379	249667001	0.00197 and 0.00531	0.00379 and 0.00482
10	1201828770	1642	08-MAY-09	0.531	0.878	1.85	0.0276	228835001	0.0504 and -0.00507	0.0276 and 0.0119
11	1201831011	1676	12-MAY-09	0.447	0.878	0.949	0.0105	228983001	-0.00581 and 0.00904	0.0105 and 0.0116
12	1201880380	1666	17-JUL-09	0.856	0.878	0.283	0.0032	232921002	0.00353 and 0.00209	0.0032 and 0.00395
13	1201888030	1688	31-JUL-09	0.739	0.878	0.224	0.0923	234008001	0.605 and 0.636	0.0923 and 0.103
14	1201925017	1670	22-SEP-09	0.557	0.878	0.624	0.0151	236858001	0.0151 and -0.00804	0.0151 and 0.022
15	1201957829	1640	06-NOV-09	0.776	0.878	0.817	0.00436	239887001	0.00356 and 0.0127	0.00436 and 0.0103

Sample Summary

Overall, the chemical yields for the analytical processes were greater than the minimum yields required in the SOW. Those not meeting the yield requirements are further discussed in the **Observation** section of this report.

OBSERVATIONS

Urine:

Americium:

One Americium-241 MB is denoted as an outlier; however, the result is less than the CL.

Out of two hundred and thirty-two Americium-241 high LCSs, one (0.43%) is greater than 125%.

Out of two hundred and thirty-one Americium-241 Low LCSs, twenty –five (10.82%) are less than 75%, and fifty-four (23.38%) are greater than 125%.

There is one less Am241 Low LCS's due to sample 1201886112 being lost during the prep phase.

Out of two thousand one hundred and eight Americium 243 yields, twenty-nine are denoted as outliers. Thirteen (0.62%) are less than the low yield of 40%.

One Americium-243 High LCS did not have a recovery because it was accidentally spiked with Am-241 LCS and is on the Am-241 graph. That point was excluded from both the database and the graph to show a better representation of the data points.

Out of thirty-five Americium 243 Low LCS, one Americium-243 Low LCS was excluded from the graph and data results due to being an outlier. Five (14.29%) are less than 75%. Six (17.14%) are greater than 125%.

Out of two hundred and seventeen Cm-243/244 yields, two are denoted as outliers; however, the results are greater than the minimum yield of 20%.

Curium:

One Curium-242 MB is denoted as an outlier; however, the result is less than the CL.

Out of eighty-two Curium 243/244 LCS high, one (1.22%) is greater than 125%.

Four Curium-243/244 MBs are denoted as outliers; however, the results are less than the CL.

Neptunium:

Out of thirty-seven Neptunium-237 yields, one (2.70%) is less than the low yield of 40%.

Out of eight Neptunium-237 low LCSs, two (25%) are greater than 125%.

Plutonium:

Three Plutonium-238 MBs are denoted as outliers; however, the results are less than the CL.

Four Plutonium-239/240 MBs are denoted as outliers; however, the results are less than the CL.

Out of five hundred and seventy-three Plutonium-239/240 high LCSs, two are denoted as outliers; however, one (0.17%) is less than 75%.

Out of five hundred and seventy-three Plutonium-239/240 Low LCSs, one is denoted as an outlier. Seventy-nine (13.79%) are less than 75%, and ninety-four (16.40%) are greater than 125%.

Out of five thousand eight hundred and thirty-nine Plutonium-242 Yields, seventy-one are denoted as outliers on the graph from April 1, 2009 til December 31, 2009. Forty-one (0.702%) are less than 50%, and one (0.00017%) is less than 25%. On the second graph of the Plutonium-242 yields from January 1, 2010 til March 31, 2010, twenty points are denoted as outliers. Thirty-seven (0.634%) are less than the low of 50%, but greater than 25%. In summation for the Pu-242 Yields: seventy-eight (1.34%) results are less than 50%, and one (0.00017%) is less than 25%.

Strontium:

Three Strontium-90 MBs are denoted as outliers; however, the results are less than the Lc.

Out of two hundred and twenty-seven Strontium-90 high LCSs, four are denoted as outliers. One (0.44%) is less than 75%, and four (1.76%) are greater than 125%.

Out of two hundred and twenty-seven Strontium-90 Low LCSs, four are denoted as outliers. Three (1.32%) are less than 75%, and eleven (4.85%) are greater than 125%.

Out of two thousand one hundred and fifty-two Strontium-90 yields, thirty-four are denoted as outliers. Sixty-five (3.02%) are less than the low yield of 50%, but greater than the minimum of 25%. Seven (0.33%) are less than the minimum of 25%. Samples 232272009, 1201868137, 1201868138, and 1201868139 failed tracer yields due to analyst error. A portion of the samples were lost during the column elution process of prep. The results are reported with an FA qualifier. Samples 231959002, 232109001, 232352001, 1201863868, and 1201863870 did not meet tracer recovery yields due to prep analyst error. The results are reported with an FA qualifier. Sample 1202015045 had a yield less than the minimum, but the LCS recovery for the nominal was within spec, so the result was reported. Sample 1202024467 had a yield less the minimum; however, the yield was on a low LCS. The recovery was as expected. The result was reported.

Total Strontium:

Out of four hundred and eleven Total Strontium yield, eight are denoted as outliers. Seventeen (4.14%) are less than the low yield of 50%.

Tritium:

One Tritium MB is denoted as an outlier; however, the result is less than CL.

Total Uranium:

Uranium-238 (ICMPS) is also referred to as Total Uranium in this QC package.

Up until August of 2009, a 5.0 ug/L Total Uranium spike was used for the Low range LCS, which was not low enough. The low range LCSs prior to August are found on the high range LCS graph.

Out of one hundred and forty-eight high Total Uranium LCSs, two (1.35%) are denoted as outliers and are less than 75%.

Out of one hundred and forty- seven Total Uranium Low LCSs, two (1.36%) are less than 75%. Twenty-two are greater than 125%.

Thorium:

One Thorium-228 yield is denoted as an outlier; however, the result is less than the CL.

The Thorium-234 yields graph is labeled as Thorium-228.

Out of seventy-three Thorium-228 yields, one (1.37%) is denoted as an outlier and is less than 40%

Uranium:

Out of one thousand two hundred and fifty-six Uranium-232 Yields, twenty-three are denoted as outliers. Thirty-two (2.55%) are less than the low yield of 40%. Five (0.40%) are greater than 125%.

Three Uranium-233/234 MB's are denoted as outliers. Eleven Uranium-233/234 MBs are greater than the RDL. Ten of the results were less than the MDA, and were reported. One, 1201889790, was greater than the MDA; however, it was notated that synthetic urine was used to prep the QCs (MB and LCSs) causing the elevated uranium counts. The results were reported.

Four Uranium-238 MBs are denoted as outliers. Eight Uranium-238 MBs are greater than the RDL. Seven of the results are less than the MDA, and were reported. One, 1201889790, was greater than the MDA; however, it was notated that synthetic urine was used to prep the QC's (MB and LCSs) causing the elevated uranium counts. The results were reported.

Out of one hundred and fifty Uranium-238 High LCSs, one is denoted as an outlier. One (0.67%) result is less than 75%, and one (0.67%) is greater than 125%.

Out of one hundred and fifty one Uranium-238 Low LCSs, two are denoted as outliers. Twenty-five (1.32%) results are less than 75%, fifty-eight (38.41%) are greater than 125%. There is one less Uranium-238 Low LCS due to one low LCS's eluate, 1201856037, was not collected during the column prep phase.

Uranium-236:

No observations were noted for this year.

Fecal:

Americium:

One Americium-241 MB was denoted as an outlier. The matrix blank, 1201924767, was greater than the required detection limit; however, the blank result is less than 5% of the least active sample in the batch and therefore is reported.

Out of one hundred and sixty-one Americum-243 Yields, four are denoted as outliers. Two (1.24%) are less than the low yield of 40%.

Out of thirty-one Americium-241 high LCSs, one (3.23%) is greater than 125%. Three high LCS's are excluded from the batch due to being P.E. samples.

Plutonium:

One Plutonium-238 MB is denoted as an outlier; however, the result is less than the CL.

One Plutonium-239/240 MB was denoted as an outlier. The matrix blank, 1201924771, did not meet the required detection limit; however, the blank result was less than 5% of the least active sample in the batch and therefore is reported. This point was excluded from the graph to show a better representation of the points.

Out of one hundred and fifty-nine Plutonium-242 yields, eleven (6.92%) are less than the low yield of 50%.

Out of eighteen Plutonium 239/240 low LCSs, one (10.06%) is less than 75%, and three (16.67%) are greater than 125%.

Incident Reports

Incident associated with Work Order 227837, April 17, 2009

Sample 227837001 (Tagword 09D0506) was accidently composited with another client's sample during prep and cannot be analyzed for U238 by ICPMS.

The incident involved sample 227837001(tagword 09D0506) for total uranium analysis and happened when compositing the sample. The technician, Genice Stewart, accidently combined an incorrect sample that was on the sample cart. The technician immediately recognized the error and reported the incident. The entire volume of sample was affected by the incident. The incident was resolved when the Group Leader of the Bioassay laboratory discussed the error and the necessity of close inspection of sample identifications with the responsible technician. The analyst was reminded to verify the sample identification prior to compositing.

This corrective action is considered closed

Incident associated with Work Order 228221, May 06, 2009

The incident involved Tagword 09D0149 which was reported as IS (Insufficient Sample) on 4/15/09 then RV (Received Valid) on 4/17/09. The laboratory performed Uranium analysis on the sample but should not have, due to the IS code on the sample.

The incident was investigated and it was found that Kacey Seagraves, Technician, received the sample and apparently scanned the container ID (9C1164072) by mistake rather than the individual tagwords. The container ID included both 09D0148 (tritium aliquot, GEL ID 228227002) and 09D0149 (Uranium aliquot) and this caused them to both be logged in with the RV code. In resolution, the Group Lead of the Bioassay laboratory has discussed the error with the employee and a refresher training was conducted on Friday, May 8, 2009. The laboratory has investigated the possibility of programming to prevent this error from occurring electronically. It is not expected to reoccur.

This corrective action is considered closed.

Incident associated with Work Order 228599, May 20, 2009

The incident involved IPS (Tagwords 09D0626) and U (09D0627) in which GEL incorrectly cancelled an analysis that resulted in a worker (Payid 73076) being denied access to his work location. The U was cancelled 4/23 by Battelle and replaced with a U238 request (09D0639). However, GEL cancelled both 09D0626 and 09D0627 in conformance with the existing company policy of grouping all analyses for a sample and treating them as one unit. Battelle uses tagwords to identify the basic ordering unit, and expects each tagword order to be treated separately. More than one tagword may be associated with a single sample, but the tagword orders are expected to be treated separately.

The incident was investigated. The RSC personnel used the "Scan Container" application. In this particular instance, Wendy Mitchell entered the individual tagword to status it; however, both tagwords were statused even though only one was entered in the "Scan Container" screen. She did not realize that when she entered an individual tagword, a List of Values (LOV) is present to choose what status code to assign the container as a whole or to an individual tagword. She assumed by typing in the tagword, it statused only the tagword she entered.

In resolution, each tagword is treated by LIMS as an individual, separate unit. The code was reverified to only update a tagword if the user enters a tagword on the following screen: http://172.16.100.101:7778/pls/lims/pm_pnnl.scan_container. The code has been updated to display the results of the database changes instead of refreshing back to the scan screen. The information displayed will communicate the number of rows changed for each of the entry types possible. The RSC has been trained to ensure that the individual tagword to assigned correctly in the future.

This corrective action is considered closed.

Incident associated with Work Order 229517, May 27, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for the Uranium analyses. Analyst Christina Kimball prepared batch 866295 containing tagword 09E0459 (GEL ID 229517001). The samples were turned into the count room and upon completion of counting it was identified that the sample and QC samples in the batch had no tracer recovery and the spiked QC samples did not have the spike. All samples were reported as Failed Analyses for Uranium. Uranium was the only analysis performed on this sample. Because this tagword was a Special Priority urine sample, all eluants were retained during initial analysis. These eluants were subsequently analyzed and the analyses still failed with no tracer recoveries. In resolution, there was a chemistry error that resulted in the loss of the samples.

This corrective action is considered closed.

Incident associated with Work Order 229353, May 28, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for the Plutonium analyses. Analyst Christina Kimball prepared batch 865446 containing tagwords 09E0061 (229353001), 09E0081 (229353002), 09E0208 (229353003), 09E0210 (229353004), 09E0275 (229353005), 09E0366 (229353006), 09E0410 (229353007) and 09E0457 (229353008). The samples were turned into the count room and upon completion of counting it was identified that the sample and QC samples in the batch had no tracer recovery and the spiked QC samples did not have the spike. All samples were reported as Failed Analyses for Plutonium. Plutonium was the only analysis requested on these samples. In resolution, there was a chemistry error that resulted in the loss of the samples.

This corrective action is considered closed.

Incident associated with Work Order 229249, May 29, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for the Plutonium analyses. Analyst Tina Schoneman prepared batch 864970 containing tagwords 09E0408 (229249001), 09E0389 (229573001), 09E0409 (229573002), 09E0432 (229573003), 09E0444 (229573004), 09E0448 (229573005), 09E0460 (229573006), 09E0471 (229573007) and 09E0472 (229573008). Tagword 09E0408 (229249001) had tritium analysis requested in addition to Plutonium. The tritium portion has been reported successfully. The samples were turned into the count room and upon completion of counting it was identified that the sample and QC samples in the batch had no tracer recovery and the spiked QC samples did not have the spike. All samples were reported as Failed Analyses for Plutonium. Plutonium was the only analysis requested on these samples. This incident report is similar to two recent reports describing the same error. The only difference was the analyst and date & time of analysis. Because a similar incident happened by two analysts, we looked further into the process verifying reagents, supplies, processes, etc. and no problems were discovered. We also looked at data for batches analyzed before, during and after the incident and all batches were performed successfully ruling out a reagent, equipment or process problem. In resolution, there was a chemistry error that resulted in the loss of the samples.

This corrective action is considered closed.

Incident associated with Work Order 229354, June 01, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for the Plutonium analyses. Analyst Tina Schoneman prepared batch 865448 containing tagwords 09E0274 (229352001), 09D0664 (229354001), 09E0049 (229354002), 09E0084 (229354003), 09E0327 (229354004), 09E0339 (229354005), 09E0445 (229354006) and 09E0456 (229354007). This batch was filtered at the same time as batch 864970 for which GEL submitted IR 5-29-09. Both of these incidents appear to have had errors at the final precipitation and filtering steps. Two different analysts had the same error in batches finalized four days apart. The samples were turned into the count room and upon completion of counting it was identified that the sample and QC samples in the batch had no tracer recovery and the spiked QC samples did not have the spike. All samples were reported as Failed Analyses for Plutonium. This batch had Sr-90 analysis on all samples as well as Pu. The Sr-90 is performed sequentially from the same aliquot. The Sr-90 results are fine. This incident report is similar to three recent reports describing the same error. The only difference was the analyst and date & time of analysis. Because a similar incident happened with two analysts, we looked further into the process verifying reagents, supplies, processes, etc. and no problems were discovered. We also looked at data for batches analyzed before, during and after the incident and all batches were performed successfully ruling out a reagent, equipment or process problem. The laboratory is in the process of adopting checklists for the analysts to use. These checklists may be used to document completion of certain prep steps required by the SOP. All analysts will be retrained to the use of the checklists and the importance of correctly performing all steps in the preparation of the samples. In resolution, there was a chemistry error that resulted in the loss of the samples.

This corrective action is considered closed.

Incident associated with Work Order 234171, August 18, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for Plutonium analysis of isotopes Plutonium-238 and Plutonium-239/240. Analyst Kacey Seagraves prepared Alpha Spec Plutonium batlch 889299 containing tagwords 09G0081 09G0494 09G0502 09G0504 09G0510 09G0516 09G0517 09G0527 09G0551. The results were reported as Failed Analysis for plutonium analysis due to failed tracer yield and LCS recovery. The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for Plutonium analysis. Tagwords 09G0558 09G0559 09G056 were reported as FA for plutonium analysis due to failed tracer yield and LCS recovery. These samples and the associated batch QC did not have tracer or LCS spike recoveries. This particular analyst was our most recently qualified analyst for this procedure. She was interviewed by the senior chemist at the time of occurance. Also the senior chemist did a method audit with the analyst and did not detect any obvious errors in the method or her technique. She was advised to document the steps in the procedure in the method check list as they were performed. We have reduced her sample load in quantity and complexity of analysis to get back up to speed.

This corrective action is considered closed.

Incident associated with Work Order 234284, August 18, 2009

The incident involved a chemistry error that resulted in a batch of samples having no tracer recoveries for Plutonium analysis. Tagwords 09G0558 09G0559 09G056 were reported as FA for plutonium analysis due to failed tracer yield and LCS recovery. These samples and the associated batch QC did not have tracer or LCS spike recoveries. This particular analyst was our most recently qualified analyst for this procedure. She was interviewed by the senior chemist at the time of occurrence. Also the senior chemist did a method audit with the analyst and did not detect any obvious errors in the method or her technique. She was advised to document the steps in the procedure in the method check list as they were performed. We have reduced her sample load in quantity and complexity of analysis to get back up to speed.

This corrective action is considered closed.

Incident associated with Work Order 234742, September 02, 2009

The incident involved a client requested check of the positive data submitted for tagwords 09G0661, 09G0665, and 09G0673. Sr hits were reported for samples 234742004, 234742005, and 234742007.

The data was checked and no errors were found. The analytical batch containing these samples had 9 total samples. The samples with hits were in position 4, 5 & 7. All other samples in the batch were below the MDA. All volumes for samples in the batch were consistent with each other. The samples were all counted on a GFPC auto detector and therefore have the same efficiency, count time, background correction, etc. The samples in question were previously recounted in house allowing an ingrowth period of 4 days for Y-90. The second counts all had higher count rates than the original which is expected for Sr-90 activity. All results when corrected for the additional ingrowth of Y-90 duplicated the original results. All batch QC was passing and as expected for the analysis. No anomalies were noted with the batch review. We have had no other abnormal Sr-90 results or concerns from other clients during this time frame in the laboratory. In summary, no errors could be identified with the data.

This incident continues on with further investigation on September 24, 2009, and is closed following the corrective actions found under September 24, 2009.

Incident associated with Work Order 234742, September 24, 2009

On September 24,2009, another incident report was submitted for work order 234742 where PNNL identified three QC samples that were in process at about the same time as the high samples from the incident regarding work Order 234742 filed on September 02, 2009 . The QC samples were all spiked at 10 dpm, but were reported as non-detects. The Pu and Am results were as expected. They requested that GEL review the chain-of-custody for the following six samples, and determine if there were opportunities for the sample fractions to get switched during the analysis process. The three 09G samples are all baselines. One person came from a power plant, one switched contractors at Hanford, and one has not been working in the nuclear industry for the last decade. Two of the people may have had opportunity for an intake, but all three are unexpected.

After further research and investigation, the evidence shows it is very likely that samples are switched between 2 batches. Batch number 892098 has 9 samples and 3 QC's. This batch contains samples 09G0661, 09G0665 and 09G0673. Batch number 892955 has 9 samples and 3 QC's. This batch contains samples 09H0120, 09H0180 and 09H0443. In both batches, the samples of concern are the 4th, 5th and 7th samples in the batch. The batches were prepped and counted one day apart and are apparently switched based on the blind spike information. On 8/25 a batch was turned into the count room labeled 892098. The paperwork for 892098 and all samples were labeled as such. On 8/26 a batch was turned into the count room and all samples were labeled 892098 but with paperwork for 892955. An assumption was made that the samples turned in on 8/25 were correct and that the samples on 8/26 were mis-labeled and therefore were re-labeled. Further investigation of the sample prep start dates and strontium separation times would have shown that 892955 should have been turned in first since that prep started on 8/19 and 892098 should have been second since that prep started on 8/20. This would have shown that it was the batch turned in first that was mislabeled and the paperwork just got switched.

The problems were identified and resolved as follows:

The initial issue was misidentified filters which were detected by the count room analyst. Previously the petri dishes which contained the filters had the sample/batch information added manually which could allow transcription errors. To resolve this issue LIMS generated labels with the sample/batch information and barcode will be printed and affixed to the filter petri dish. The sample information will be automatically confirmed when the samples are scanned to the bioassay count room after sample preparation identifying the submitted samples with the analytical batch. The second issue involved documentation. Analytical process anomalies such as unusual sample descriptions, chemical reactions, spike information, etc. are recorded by the analyst on the batch history check list. The resolution is that all issues with the batch will now be included in this check list to provide information for data reviewers/validators. Decisions on vague situations will be confirmed/resolved by the appropriate group leader or senior staff. Increased throughput has increased the number of samples per analyst.

This corrective action is considered closed.

Incident associated with Work Order 239331, November 25, 2009

The incident involved modified Am/Cm results reported for PNNL 239331 due to the analyst using an incorrect blank population when processing the results.

The Am/Cm results for the attached tagwords were modified due to the analyst processing the samples with the wrong blank population. The error was discovered after business hours last night. The error was resolved when the samples were re-processed with the correct blank population the following day. The results went slightly down. The client was informed immediately and corrected data was sent electronically.

This corrective action is considered closed.

Incident associated with Work Order 241522, November 25, 2009

The incident involved modified tritium results reported for PNNL 241522 due to an error in data processing that requires a re-calculation of the data.

The tritium results for the attached tagwords were modified due to an error in data processing that required a re-calculation of the data. As soon as the error was discovered, the samples were re-processed. The client was informed immediately and corrected data sent electronically. No further action taken.

This corrective action is considered closed.

Incident associated with Work Order 240774, December 03, 2009

The incident involved a questioning of the reported values for Tagword 09K0145, which is associated with a known past intake, and the values for both the Am and Pu results were expected to be positive. The Am was detected, but the Pu isotopes were both reported as 0.00 E+00. In the same report results for 09K0440 were positive for both Pu isotopes. The sample was a baseline, which meant it was collected before there was an exposure potential. These results in combination raise concerns about this batch, which prompted a data recheck.

After investigating, it was suspected that there may have been an error with the data. Sample 240774004 was a sample with container ID 9J1336012. Three tagwords were issued for this container. 09K0009 (Pu), 09K0010 (Am) and 09K0011 (U-238). Each tagword received its own GEL client ID and was in three different work orders 240777, 240771 and 240789 respectively. Since Americium and Plutonium are analyzed sequentially, they are shown on the same que sheet. However, because they each received different work orders they were not shown sequentially on the que sheet since the que sheet orders numerically by work order then sample ID. The prep analyst recognized this and was very deliberate in listing on the que sheet that the samples were not in numerical order. However, it is suspected that the count room analyst placed the plutonium samples into the detectors in numerical order which would have resulted in the counting source for 09K0145 Pu being placed into the detector for 09K0440 Pu. The samples were recounted as labeled and in the proper order. The Group Leader verified the placement in the detectors. The data recount confirmed that the detectors were loaded into the detector improperly. Jay MacLellan requested a discussion in the incident report of why the corrective actions for the Sr sample switch incident weren't effective for this incident. This incident is different in that the samples were labeled correctly upon delivery to the countroom. In the Sr incident, the samples were not labeled correctly upon delivery to the countroom. The corrective action for the Sr incident was in place for these samples and was effective in ensuring the samples were properly labeled. This incident was due to the count room analyst not keeping the samples in the correct order when placing the samples into the detectors. This was brought on by the samples being out of numerical order on the que sheet due to having the multiple sample ID's associated with a sequential analysis of a single sample. This incident is resolved in that we have a program in LIMS that will allow the samples to be re-ordered when these infrequent situations arise. This will allow less confusion when sample ID's are not in numerical order.

This corrective action is considered closed.

Incident associated with Work Order 245468, February 15, 2010

The incident involved a uranium batch that had no tracer recoveries for the entire batch including QC samples. An apparent analytical problem took place. Six samples are involved. Tagwords are 10A0353, 10A0359, 10A0362, 10A0381, 10A0601 and 10A0625. The samples were reported as failed analysis for Isotopic Uranium analysis. There were no analytical problems noted by the analyst and the analytical checklist is filled out completely and correctly not showing any problems. The place of error could not be pinpointed.

Analyst Kristi Williams prepped the referenced batch on 1/29/10. While Kristi Williams is our newest Analyst, she has been qualified since the first of January and all qualification requirements were met. It is suspected that the most likely cause is that a wrong reagent was used and not noticed or a step was missed and not noticed. Kristi has performed several batches to date and has not had an error like this. The group leader will continue to monitor Kristi's performance; however, it's believed that this error is due to her minimal experience. In resolution, the Group Leader, Bob Timm, has witnessed Kristi perform her evolutions and does not feel there is a process change that would be beneficial. He has gone over this error with the analyst and discussed possible places for error stressing the importance of attention to detail.

This corrective action is considered closed.

Incident associated with Work Order 241513, March 08, 2010

This incident involved a client requested recheck of Tagwords 09K0224, 10A0648 and 10A0649. The GEL sample IDs are 241513001, 244287001, and 244071001. These were for Am243.

09K0224: The analytical batch 925082 which contained tagword 09K0224 had an anomaly that may not be related to the hit but does raise a question. The batch had another sample which showed no activity nor did the batch blank show activity. The batch Laboratory Control Sample was as expected at 106% recovery. The anomaly is that the low spiked sample showed up at 0.155 dpm; however, it was spiked with 0.0208 dpm so the recovery was 745% which is very odd. In doing some research, it was found that the Am-243 batch was prepped along side a Np-237 batch. Both batches were poured up and traced at the same time using the same pipet and in the same vicinity. The concern is that the Np-237 test uses an Am-243 tracer for it's Np-239 daughter and that Am-243 tracer is at 4378 dpm/ml. If the Am-243 hit in sample 09K0224 is unexpected by PNNL, there could have been some cross contamination when prepping the batch with the Am-243 tracer used for Np-237 analysis. In conclusion for 09K0224, the activity in 09K0224 could be cross contamination in the laboratory based on the information above. This sample count had 17 net counts with a background of 3 counts. Based on the count data, the activity looks to be Am-243. It's just the source of Am-243 that is in question.

10A0648: This sample has 6 gross counts and 0 bkg for a net of 6 counts. The activity is just above the PNNL action level and a difference of 1 count less would have resulted in the result being less than the action level. The counts in the ROI do fall in the region of interest and correspond with the energy expected for Am-243.

10A0649: This sample has a result less than the PNNL action level. The gross counts are 0 and the background counts are 2 which give a negative net result. All calculations are verified to be correct and also correspond to the achieved count rates.

This incident is considered closed.

Previous Years Corrective Actions

There were no corrective actions carried over from the previous years.

APPENDIX C

QUALITY CONTROL INTERCOMPARISON PARTICIPATION RESULTS

(Historical File Only)

U.S. DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY

General Engineering Laboratories, LLC
Charleston, South Caroline

Test Identification: NRIP-09-SF
Test Radionuclides: ^{241}Am ^{241}Am, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{235}U, ^{234}U, ^{90}Sr, ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{243}Cm
Matrix Description: Synthetic Feces[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, April 1, 2009

Measurement Results

Nuclide	NIST Value[2,3]		Reported Value[4]		Difference[5]
	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	(±% Bias)
^{243}Cm	0.591	1.26	0.529	22.9	-11
^{241}Am	1.407	0.82	1.230	22.8	-13
^{238}Pu	0.477	0.71	0.416	13.8	-13
^{240}Pu	0.599	0.79	0.522	21.0	-13
^{230}Th	0.726	0.61	0.680	32.4	-6.3
^{238}U	1.591	0.63	1.264	18.1	-20
^{234}U	1.532	0.98	1.190	13.0	-22
^{235}U	0.073	0.65	0.066	40.3	-10
^{226}Ra	1.225	2.50	1.230	32.5	0.4
^{90}Sr	12.88	0.77	10.30	27.8	-20

NR= Not Reported NA= Not Applicable

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha- and Beta-Spectrometry Mass Spectrometry	Alpha, Beta, and Gamma Spectrometry

Evaluation (per ANSI N42.22 and N13.30)

Nuclide	N42.22[8]		N13.30[9]	
	ANSI N42.22 Traceable	Traceability Limit (±Percent)	Results Acceptable per N13.30 Criteria (Pass/Fail)	
			Bias	Precision
^{243}Cm	Yes	31	Pass	Pass
^{241}Am	Yes	30	Pass	Pass
^{238}Pu	Yes	18	Pass	Pass
^{240}Pu	Yes	27	Pass	Pass
^{230}Th	Yes	45	Pass	Pass
^{238}U	Yes	22	Pass	Pass
^{234}U	No	15	Pass	Pass
^{235}U	Yes	54	Pass	Pass
^{226}Ra	Yes	49	Pass	Pass
^{90}Sr	Yes	30	Pass	Pass

Samples Distributed August 12, 2009
Reporting Data Received October 15, 2009

For the Director

Michael Unterweger,
Group Leader
Radioactivity Group
Physics Laboratory

(Continued)

U.S. DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY

General Engineering Laboratories, LLC
Charleston, South Caroline

Test Identification: NRIP-09-SF
Test Radionuclides: ^{241}Am, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{235}U, ^{234}U, ^{90}Sr, ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{243}Cm
Matrix Description: Synthetic Feces[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, April 1, 2009

Measurement Results

Nuclide	NIST Value [2,3]		Reported Value[4]		Difference[5]
	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	Massic Activity Bq•g^{-1}	Relative Expanded Uncertainty (%, k=2)	(±% Bias)
^{243}Cm	0.591	1.26	0.577	13.6	-2.3
^{241}Am	1.407	0.82	1.368	11.4	-2.7
^{238}Pu	0.477	0.71	0.438	14.0	-8.2
^{240}Pu	0.599	0.79	0.568	14.2	-5.0
^{230}Th	0.726	0.61	0.702	13.9	-3.2
^{238}U	1.591	0.63	1.344	14.3	-15
^{234}U	1.532	0.98	1.320	12.5	-14
^{235}U	0.073	0.65	0.059	48.1	-19
^{226}Ra	1.225	2.50	1.23	36.5	-5.5
^{90}Sr	12.88	0.77	11.58	11.8	-10
^{137}Cs	265.4	0.72	267.4	10.4	0.7
^{60}Co	271.4	0.54	280.6	10.6	3.4
^{57}Co	67.56	1.77	70.3	12.1	4.0

NR= Not Reported NA= Not Applicable

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha- and Beta-Spectrometry Mass Spectrometry	Alpha, Beta, and Gamma Spectrometry

Evaluation (per ANSI N42.22 and N13.30)

Nuclide	N42.22[8]		N13.30[9]	
	ANSI N42.22 Traceable	Traceability Limit (±Percent)	Results Acceptable per N13.30 Criteria (Pass/Fail)	
			Bias	Precision
^{243}Cm	Yes	20	Pass	Pass
^{241}Am	Yes	17	Pass	Pass
^{238}Pu	Yes	19	Pass	Pass
^{240}Pu	Yes	20	Pass	Pass
^{230}Th	Yes	20	Pass	Pass
^{238}U	Yes	18	Pass	Pass
^{234}U	Yes	16	Pass	Pass
^{235}U	Yes	58	Pass	Pass
^{226}Ra	Yes	52	Pass	Pass
^{90}Sr	Yes	16	Pass	Pass
^{137}Cs	Yes	16	Pass	Pass
^{60}Co	Yes	16	Pass	Pass
^{57}Co	Yes	19	Pass	Pass

Samples Distributed August 12, 2009
Reporting Data Received October 15, 2009

For the Director

Michael Unterweger,
Group Leader
Radioactivity Group
Physics Laboratory

(Continued)

As guidance for the proper use of this Report, it should be emphasized that the National Institute of Standards and Technology is concerned only with fostering good measurement capability and consistency with the national measurements system. The assurance of the proper application of that capability to the ultimate consumer products is the responsibility of each manufacturer and of the Federal regulatory agencies.

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Notes

(1a) Five test-samples and three (identical matrix) blanks were provided for this test. Each sample consisted of approximately 100 grams of synthetic fecal material contained in a plastic zip-lock bag that was packed in a plastic container.

Composition of the Synthetic Feces

Reagent	g/sample
Calcium Nitrate	0.97
Ferric Ammonium Sulfate	0.04
Magnesium Carbonate	0.61
Potassium Carbonate	0.83
Ammonium Dihydrogen Phosphate	2.1
Sodium Sulfate	0.37
Ammonium Chloride	0.04
Zinc Sulfide	0.01
Stannous Chloride	0.03
Leucine	7.1
Lysine	5.1
Methionine	0.8
Threonine	2
Palmitic Acid	3
Stearic Acid	2
Cellulose	4
Gelatin	5
Oleic Acid (Liquid)	1
Peanut Oil	1.5
Water (distilled)	65

(1b) The test samples were prepared by depositing a known amount of a NIST calibrated "spike" solution (aqueous solution containing known quantities of $^{241}Am^{241}Am$, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{235}U, ^{234}U, ^{90}Sr, ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, and ^{243}Cm to the center of individual ashless paper filters (37 mm diameter). After deposition of this solution, filters were dried overnight. Once dry, each filter was sandwiched between two unspiked filters. Each sandwich was then slipped into a low-density polyethylene sleeve (wall density ~0.1 mm) and sealed for counting. After confirmation measurement, each spiked filter pack was placed inside of the matrix contained plastic zip-lock bag (1a) for packaging and shipment.

(2a) Solutions of tracers were prepared by gravimetric dilutions of NIST Standard Reference Material SRM's or NIST calibrated solutions. The dilution factors at each step were confirmed by radioactivity measurements.

(2b) The analysis methodology and nomenclature used for the NIST uncertainties are based on uniform guidelines [cf., B.N. Taylor and C. E. Kuyatt, NIST Technical Note 1297 (1994)] and are compatible with those adopted by the principal international metrology standardization bodies. Individual uncertainties have the significance of one standard deviation of the mean, or an approximation thereof. The relative combined uncertainty is the quadratic combination of the standard

deviation (or standard deviation of the mean where appropriate), or approximation thereof, for the following component uncertainties:

	Nuclide (SRM Identification)	Uncertainty (%, 1s)
a)	^{57}Co(NIST calibration)	1.77
b)	^{60}Co(4915F)	0.25
c)	^{137}Cs(4233D)	0.34
d)	^{90}Sr(4919H)	0.37
e)	^{210}Po(4337)	2.5
f)	^{210}Pb(4337)	2.5
g)	^{226}Ra(4966)	0.44
h)	^{234}U(4321C)	0.49
i)	^{235}U(4321C)	0.31
j)	^{238}U(4321C)	0.30
k)	^{238}Pu(4323B)	0.34
l)	^{240}Pu(4338A)	0.38
m)	^{241}Am(4322B)	0.48
n)	^{243}Cm (4329)	0.47
i)	Gravimetrics (dilutions)	0.05

The individual certified uncertainties of standard reference materials are based on the quadratic combination of all sources of uncertainty manifested in the preparation of the material. These uncertainties may result from uncertainties from any or all of the following: alpha-decay emission rate, background, balance calibration, decay corrections, decay-scheme data, extrapolation of alpha-particle-count-rate-versus-energy to zero energy, live time, alpha-particle detection efficiency, alpha-emitting impurities, gamma-emitting impurities.
The **Relative Expanded Uncertainty** is obtained by multiplying the standard uncertainty by a coverage factor of **k=2** and is assumed to provide an uncertainty interval of approximately 95 percent confidence.

(3) Half-lives used

	Nuclide	Half-life
a)	^{57}Co	271.79±0.09 d
b)	^{60}Co	5.2714±0.0005 y
c)	^{90}Sr	28.78±0.04 y
d)	^{137}Cs	30.07+0.03 y
e)	^{210}Po	138.376±0.002 d
f)	^{210}Pb	22.20±0.22 y
g)	^{226}Ra	1600±7 y
h)	^{230}Th	(7.538±0.030) x 10^4 y
h)	^{234}U	(2.455±0.006) x 10^5 y
i)	^{235}U	(7.038+0.005) x 10^8 y
j)	^{238}U	(4.468±0.003) x 10^9 y
k)	^{238}Pu	87.74±0.04 y
l)	^{240}Pu	6564±11 y
m)	^{241}Am	432.2±0.5 y
n)	^{243}Cm	28.5±0.2 y

Note: Half-life data are based on NIST certificates (Note 2b) or Evaluated Nuclear Structure Data File (ENSDF 2009). Uncertainties quoted at one sigma level.

(4) The laboratory value represents the mean of five replicate measurements. The reported uncertainty was multiplied by a coverage factor of **k=2**.

(5) The **Difference** quoted is the difference between the **NIST Value** and **Reported Value**, expressed as a percent relative to the **NIST Value**.

(6) Test samples were prepared by gravimetric dilutions of NIST calibrated solutions and SRM's. These solutions and SRM's were calibrated using the following activity measurement methodologies:

Nuclide	Methodology

a)	^{60}Co	Pressurized "4π" γ ionization chamber "A" calibrated using a cobalt-60 solution whose activity was determined by "4π"-(β+γ)-coincidence and anti-coincidence counting
b)	^{57}Co	Pressurized "4π" γ ionization chamber "A" calibrated using a cobalt-60 solution whose activity was determined by "4π"-(β+γ)-coincidence and anti-coincidence counting
c)	^{90}Sr	NIST 4πβ liquid-scintillation counting system
d)	^{137}Cs	Pressurized "4π"-γ-ionization chamber "A" calibrated using a cesium-137 solution whose activity was determined by "4π"-(e + X)-γ-anti-coincidence counting Pressurized "4π" gamma ionization chamber "A" calibrated using a barium-133 solution whose number of cesium-137 atoms was determined by isotope-dilution mass spectrometry
e)	$^{210}Po(^{210}Po)$	4παβ liquid-scintillation counting system
f)	^{210}Pb	4παβ liquid-scintillation counting system
g)	^{226}Ra	Pressurized "4π" γ ionization chamber "A"
h)	^{230}Th	Two 4πα liquid scintillation counting systems
i)	^{234}U, ^{235}U, ^{238}U	Mass spectrometry, silicon surface barrier alpha-detection, and 4π (α+β) liquid-scintillation counting systems
j)	^{238}Pu	NIST "0.1π"α defined-solid-angle scintillation detector Two 4πα liquid scintillation counting systems
k)	^{240}Pu	Two 4πα liquid scintillation counting systems
l)	^{241}Am	4πα liquid-scintillation counting system
m)	^{243}Cm	NIST "0.8π" alpha and "0.1π" alpha defined-solid-angle counters with scintillation detectors

(7) Summary of the reporting laboratory methodologies.

(8) ANSI N42.22 defines the acceptance criteria for verification testing by NIST as:

$$|V_R - V_N| < 3 * \sqrt{u_c^2(N) + u_c^2(R)}$$

Where: V_N = NIST Value;
V_R = Reported Value;
$u_c(N)$= standard combine uncertainty of the NIST value, V_N;
$u_c(R)$= standard combine uncertainty of the Laboratory value, V_R; and

$3 * \sqrt{u_c^2(N) + u_c^2(R)}$ = Traceability Limit (limit to which measurement traceability may be claimed with 99% confidence)

(9) ANSI N13.30 defines criteria for acceptable bias between -25 and +50 percent, and acceptable precision between -40 and +40 percent, 1 sigma total propagated uncertainty.

Reference:

ANSI National Standards Institute, ANSI N42.22-1995, "Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control."

ANSI National Standards Institute, ANSI N13.30-1996, "Performance Criteria for Radiobioassay."

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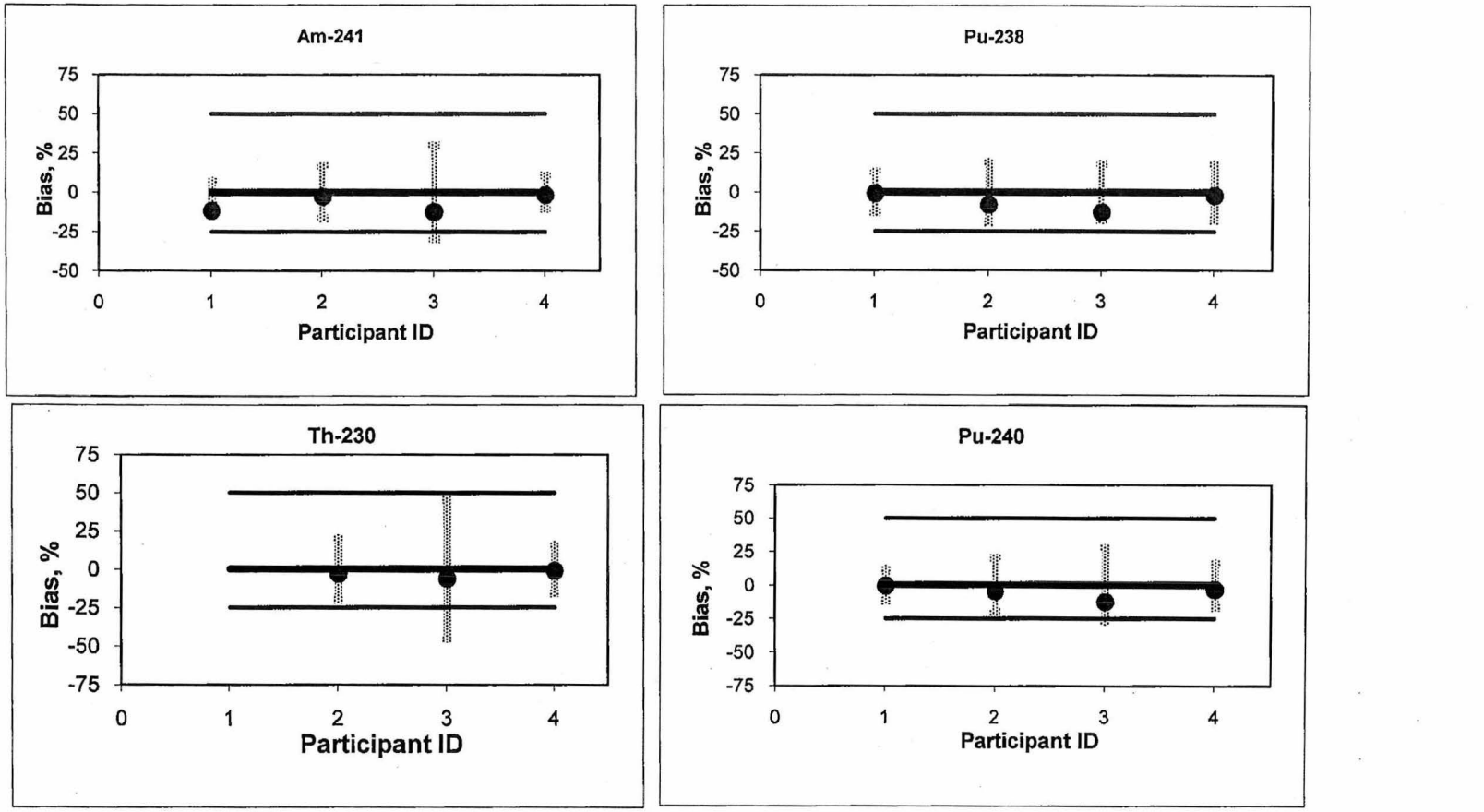
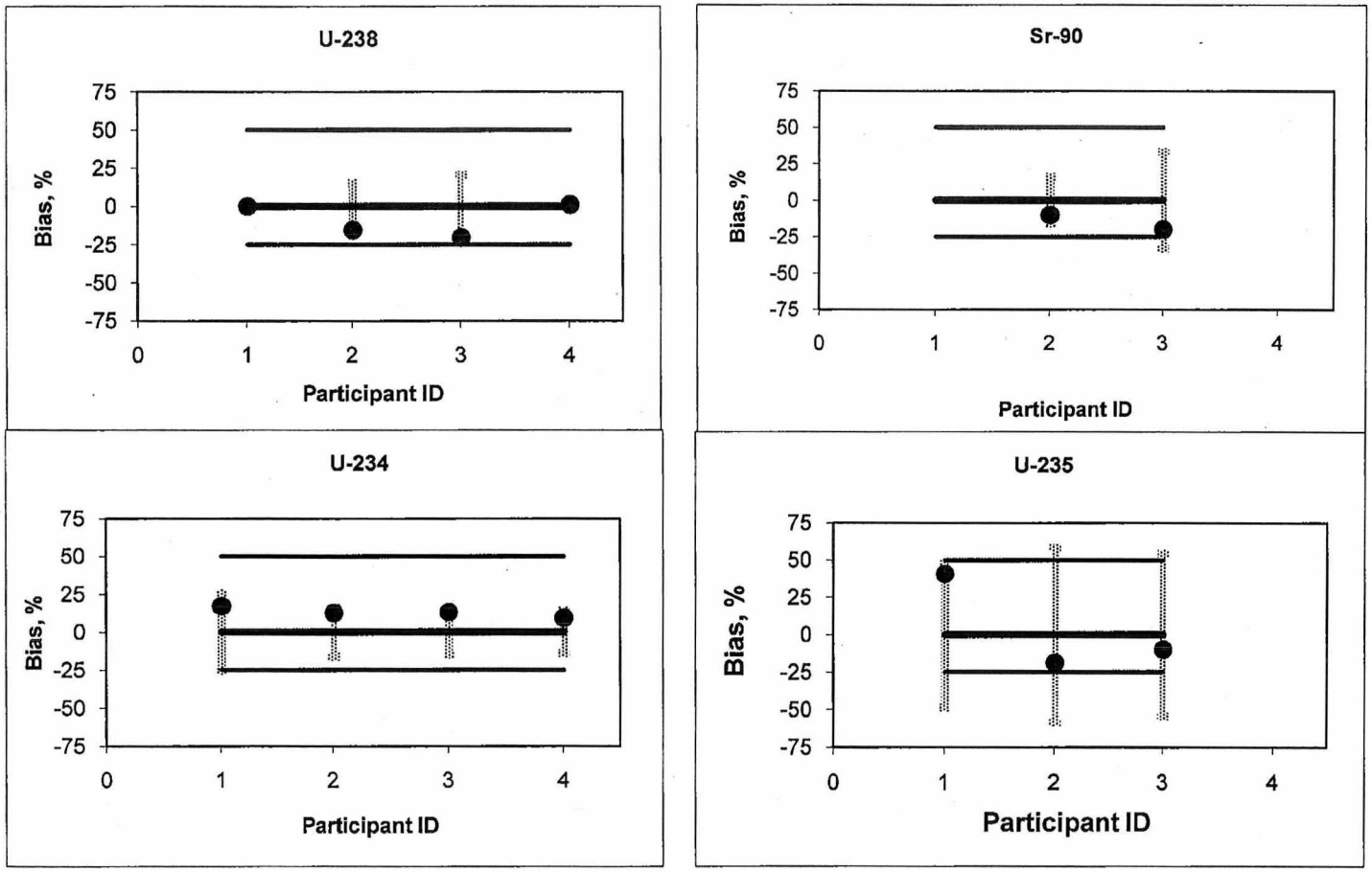
Ms. Svetlana Nour (301) 975-4927 Svetlana.nour@nist.gov

NIST Radiochemistry Intercomparison Program NRIP-2009

Distribution of Reported Results for

^{241}Am, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{234}U, ^{235}U, ^{210}Po, ^{226}Ra, ^{243}Cm, ^{210}Pb, ^{90}Sr, ^{60}Co, ^{57}Co, ^{137}Cs in Synthetic Feces Samples

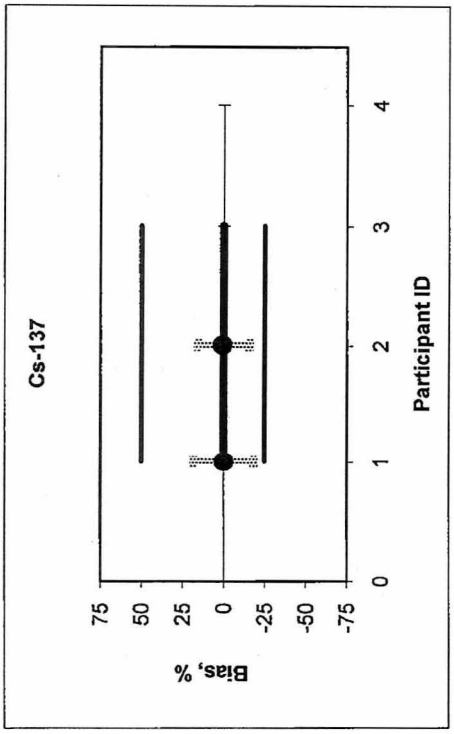


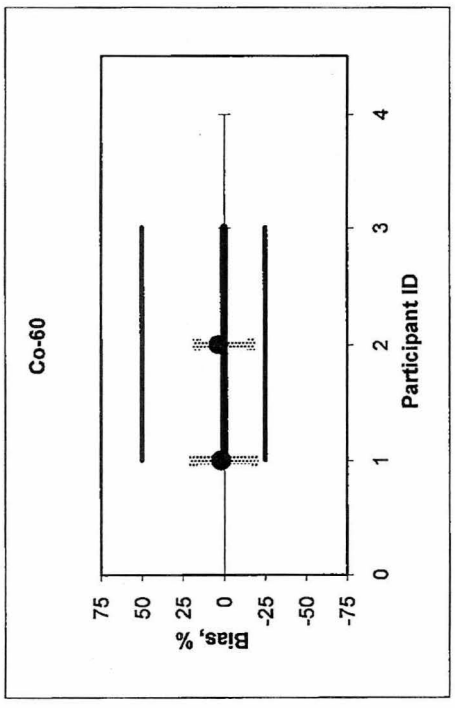


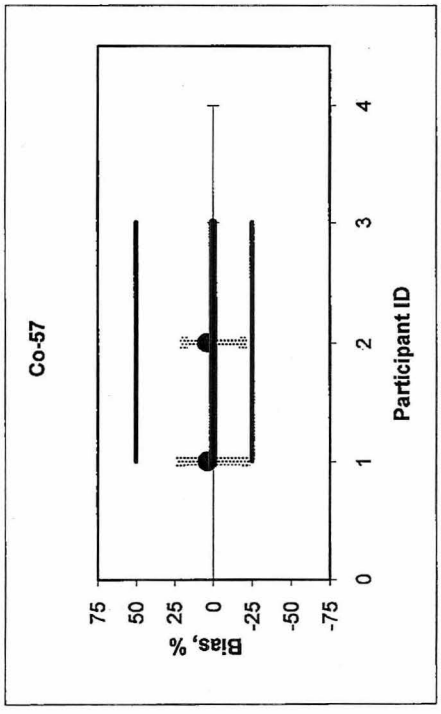
Note: Uncertainty bars represent acceptance criteria defined by ANSI N42.22.
Participant ID:

GEL 2 and 3

700


Cs-137
Bias, %
75
50
25
0
-25
-50
-75
0
1
2
3
4
Participant ID


Co-60
Bias, %
75
50
25
0
-25
-50
-75
0
1
2
3
4
Participant ID


Co-57
Bias, %
75
50
25
0
-25
-50
-75
0
1
2
3
4
Participant ID

U.S. DEPARTMENT OF COMMERCE

National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY

General Engineering Laboratories, LLC
Charleston, South Carolina

Test Identification: NRIP '09-SU (Set #1)
Analytes: ^{57}Co, ^{60}Co, ^{90}Sr, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{230}Th, ^{234}U, ^{235}U, ^{238}U, ^{238}Pu, ^{240}Pu, ^{241}Am, ^{243}Cm
Matrix Description: Synthetic Urine[1]
Test Activity Range: 0.01 Bq · sample^{-1} to 250 Bq · sample^{-1}
Reference Time: 12:00 EST, September 1, 2009

Measurement Results

Nuclide	NIST Value [2,3]		Reported Value[4]		Difference[5]
	Massic Activity Bq · g^{-1}	Relative Expanded Uncertainty (%, k=2)	Massic Activity Bq · g^{-1}	Relative Expanded Uncertainty (%, k=2)	(%)
^{57}Co	129	1.72	140	11.2%	8.7%
^{60}Co	724	0.54	750	10.4%	3.6%
^{90}Sr	35.9	0.78	39.3	11.7%	9.5%
^{137}Cs	740	0.73	759	10.3%	2.6%
^{210}Po	16.1	3.22	14.22	10.7%	-11.8%
^{226}Ra	3.46	0.89	3.20	32.6%	-7.7%
^{230}Th	2.04	0.61	1.98	13.3%	-3.3%
^{234}U	4.32	1.00	3.77	11.8%	-12.6%
^{235}U	0.206	0.65	0.212	41.0%	3.0%
^{238}U	4.48	0.63	3.91	17.9%	-12.7%
^{238}Pu	1.34	0.71	1.32	13.9%	-1.3%
^{240}Pu	1.69	0.79	1.68	14.6%	-0.5%
^{241}Am	3.96	0.82	3.73	12.4%	-5.8%
^{243}Cm	1.67	1.04	1.50	13.4%	-10.0%

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha-, Beta- and Gamma-Spectrometry, Mass Spectrometry	Alpha-, and Gamma-Spectrometry, Gas Flow Proportional Counting, Alpha Scintillation Counting (Lucas Cell)

Evaluation (per ANSI N42.22 and N13.30)

Nuclide	N42.22[8]		N13.30[9]	
	ANSI N42.22 Traceable	Traceability Limit (%)	Results Acceptable per N13.30 Criteria (Pass/Fail)	
			Bias	Precision
^{57}Co	Yes	18.5%	Passed	Passed
^{60}Co	Yes	16.2%	Passed	Passed
^{90}Sr	Yes	19.2%	Passed	Passed
^{137}Cs	Yes	15.9%	Passed	Passed
^{210}Po	Yes	15.0%	Passed	Passed
^{226}Ra	Yes	45.2%	Passed	Passed
^{230}Th	Yes	19.3%	Passed	Passed
^{234}U	Yes	15.6%	Passed	Passed
^{235}U	Yes	63.3%	Passed	Passed
^{238}U	Yes	23.4%	Passed	Passed
^{238}Pu	Yes	20.6%	Passed	Passed
^{240}Pu	Yes	21.9%	Passed	Passed
^{241}Am	Yes	17.6%	Passed	Passed
^{243}Cm	Yes	18.2%	Passed	Passed

Samples Distributed: September 28, 2009
Reporting Data Received: November 25, 2009

For the Director

Michael P. Unterweger,
Group Leader
Radioactivity Group
Physics Laboratory

(continued)

U.S. DEPARTMENT OF COMMERCE

National Institute of Standards and Technology
Gaithersburg, MD

REPORT OF TRACEABILITY

General Engineering Laboratories, LLC
Charleston, South Carolina

Test Identification: NRIP '09-SU (Set #2)
Analytes: ^{57}Co, ^{60}Co, ^{90}Sr, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{230}Th, ^{234}U, ^{235}U, ^{238}U, ^{238}Pu, ^{240}Pu, ^{241}Am, ^{243}Cm
Matrix Description: Synthetic Urine[1]
Test Activity Range: 0.01 Bq · sample^{-1} to 250 Bq · sample^{-1}
Reference Time: 12:00 EST, September 1, 2009

Measurement Results

Nuclide	NIST Value [2,3]		Reported Value[4]		Difference[5]
	Massic Activity Bq · g^{-1}	Relative Expanded Uncertainty (%, k=2)	Massic Activity Bq · g^{-1}	Relative Expanded Uncertainty (%, k=2)	(%)
^{57}Co	129	1.72	133	17.0%	2.9%
^{60}Co	724	0.54	722	12.5%	-0.3%
^{90}Sr	35.9	0.78	36.6	11.3%	1.8%
^{137}Cs	740	0.73	725	14.8%	-2.1%
^{210}Po	16.1	3.22	14.12	10.3%	-12.4%
^{226}Ra	3.46	0.89	3.09	34.6%	-10.9%
^{230}Th	2.04	0.61	1.99	10.8%	-2.7%
^{234}U	4.32	1.00	4.05	17.0%	-6.1%
^{235}U	0.206	0.65	0.206	40.8%	-0.2%
^{238}U	4.48	0.63	4.28	16.4%	-4.6%
^{238}Pu	1.34	0.71	1.28	16.1%	-4.6%
^{240}Pu	1.69	0.79	1.65	12.0%	-2.3%
^{241}Am	3.96	0.82	3.56	21.4%	-10.0%
^{243}Cm	1.67	1.04	1.46	25.3%	-12.3%

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha-, Beta- and Gamma-Spectrometry, Mass Spectrometry	Alpha-, and Gamma-Spectrometry, Gas Flow Proportional Counting, Alpha Scintillation Counting (Lucas Cell)

Evaluation (per ANSI N42.22 and N13.30)

Nuclide	N42.22[8]		N13.30[9]	
	ANSI N42.22 Traceable	Traceability Limit (%)	Results Acceptable per N13.30 Criteria (Pass/Fail)	
			Bias	Precision
^{57}Co	Yes	26.3%	Passed	Passed
^{60}Co	Yes	18.7%	Passed	Passed
^{90}Sr	Yes	17.3%	Passed	Passed
^{137}Cs	Yes	21.8%	Passed	Passed
^{210}Po	Yes	14.4%	Passed	Passed
^{226}Ra	Yes	46.3%	Passed	Passed
^{230}Th	Yes	15.8%	Passed	Passed
^{234}U	Yes	24.0%	Passed	Passed
^{235}U	Yes	61.1%	Passed	Passed
^{238}U	Yes	23.4%	Passed	Passed
^{238}Pu	Yes	23.0%	Passed	Passed
^{240}Pu	Yes	17.7%	Passed	Passed
^{241}Am	Yes	29.0%	Passed	Passed
^{243}Cm	Yes	33.3%	Passed	Passed

Samples Distributed: September 28, 2009
Reporting Data Received: November 25, 2009

For the Director

Michael P. Unterweger,
Group Leader
Radioactivity Group
Physics Laboratory

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Notes

(1a) Five test-samples and three (identical matrix) blanks were provided for this test. Each sample consisted of approximately 1000 grams of synthetic urine material contained in a 1L polyethylene bottle.

Composition of the Synthetic Urine Matrix:

Reagent Name	CAS#	Chemical formula	wt.% (g/g)
Oxalic Acid Dihydrate	6153-56-6	$C_2H_2O_4 \times 2H_2O$	0.00190%
Pepsin	9001-75-6	N/A	0.00275%
Lactic Acid (liquid)	50-21-5	$C_3H_6O_3$	0.00891%
Magnesium Sulfate Heptahydrate	10034-99-8	$MgSO_4 \times 7H_2O$	0.0436%
Glucose, D(+)	50-99-7	$C_6H_{12}O_6$	0.0455%
Citric Acid (anhydrous)	77-92-9	$C_6H_8O_7$	0.0512%
Calcium Chloride Dihydrate	10035-04-8	$CaCl_2 \times 2H_2O$	0.0597%
Hippuric Acid	495-69-2	$C_9H_9NO_3$	0.0597%
Sodium Metasilicate Nonahydrate	13517-24-3	$Na_2SiO_3 \times 9H_2O$	0.00673%
Ammonium Chloride	12125-02-9	NH_4Cl	0.101%
Creatine Monohydrate	6020-87-7	$C_4H_9N_3O_2 \times H_2O$	0.104%
Sodium Chloride	7647-14-5	NaCl	0.220%
Sodium Phosphate Monobasic Monohydrate	10049-21-5	$NaH_2PO_4 \times H_2O$	0.259%
Potassium Chloride	7447-40-7	KCl	0.325%
Sodium Sulfate (anhydrous)	7757-82-6	Na_2SO_4	0.409%
Urea (Carbamide)	57-13-6	CH_4N_2O	1.52%
Nitric Acid 70 wt.% (~50mL/L soln.)	7697-37-2	HNO_3	6.70%
Distilled Water		H_2O	90.1%

(1b) Test samples were prepared by adding a known amount of a NIST calibrated spike solution (aqueous solution containing well known quantities of ^{57}Co, ^{60}Co, ^{90}Sr, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{230}Th, ^{234}U, ^{235}U, ^{238}U, ^{238}Pu, ^{240}Pu, ^{241}Am and ^{243}Cm) into each bottle containing the synthetic urine matrix. After preparation, sample bottles were counted individually to confirm the added activity.

(2a) Solutions added to the synthetic urine samples were prepared by gravimetric dilutions of NIST Standard Reference Material SRM's or NIST calibrated solutions. The dilution factors at each step were confirmed by radioactivity measurements.

(2b) The analysis methodology and nomenclature used for the NIST uncertainties are based on uniform guidelines [cf., B.N. Taylor and C. E. Kuyatt, NIST Technical Note 1297 (1994)] and are compatible with those adopted by the principal international metrology standardization bodies. Individual uncertainties have the significance of one standard deviation of the mean, or an approximation thereof. The relative combined uncertainty is the quadratic combination of the standard deviation (or standard deviation of the mean where appropriate), or approximation thereof, for the following component uncertainties:

	Nuclide /(SRM Identification)	Standard Combined Uncertainty (%)
a)	^{57}Co /(NIST calibrated)	1.85
b)	^{60}Co /(4915F)	0.25
c)	^{90}Sr /(4919H)	0.37
d)	^{137}Cs /(4233D)	0.34
e)	^{210}Pb /(4337)	1.2
f)	^{226}Ra /(4966)	0.40
g)	^{230}Th /(4342)	0.29
h)	^{234}U /(4321C)	0.49
i)	^{235}U /(4321C)	0.31
j)	^{238}U /(4321C)	0.30
k)	^{238}Pu /(4323B)	0.34
l)	^{240}Pu/(4338A)	0.38
m)	^{241}Am/(4322B)	0.32
n)	^{243}Cm /(4329)	0.47
	Other Sources	
o)	gravimetric (dilutions)	0.1
p)	half life (decay corrections)	0.07(^{57}Co), 0.08(^{90}Sr), 0.09(^{137}Cs), 0.21(^{243}Cm)
q)	ingrowth	0.77 (^{210}Pb), 1.5 (^{210}Po)

The individual certified uncertainties of standard reference materials are based on the quadratic combination of all sources of uncertainty manifest in the preparation of the material. These uncertainties may result from uncertainties from any or all of the following: alpha-decay emission rate, background, balance calibration, decay corrections, decay-scheme data, extrapolation of alpha-particle-count-rate-versus-energy to zero energy, live time, alpha-particle detection efficiency, alpha-emitting impurities, and gamma-emitting impurities.

The **Relative Expanded Uncertainty** is obtained by multiplying the combined uncertainty by a coverage factor of **k=2** and is assumed to provide an uncertainty interval of approximately 95 percent confidence.

(3) Half-Lives Used (Uncertainties are quoted at a one-sigma level.)

Nuclide		Half-Life (days, d, or years, y)*
a)	^{57}Co	271.80 ± 0.05 d
b)	^{60}Co	5.2710 ± 0.0008 y
c)	^{90}Sr	28.80 ± 0.07 y
d)	^{137}Cs	30.05 ± 0.08 y
e)	^{210}Pb	22.23 ± 0.12 y
f)	^{210}Po	138.3763 ± 0.0017 d
g)	^{226}Ra	1600 ± 7 y
h)	^{230}Th	75400 ± 300 y
i)	^{234}U	(2.455 ± 0.006) x 10^5 y
j)	^{235}U	(7.04 ± 0.01) x 10^8 y
k)	^{238}U	(4.468 ± 0.005) x 10^9 y
l)	^{238}Pu	87.74 ± 0.03 y
m)	^{240}Pu	6561 ± 7 y
n)	^{241}Am	432.6 ± 0.6 y
o)	^{243}Cm	29.1 ± 0.1 y

*Half-life data are based mainly on the *Table of Radionuclides, Vol. 1, 2, 3 & 4*, M.M. Bé, et al., Bureau International des Poids et Measures, Pavillon de Breteuil F-92312 Sèvres Cedex FRANCE (2004-2008), available at:
http://www.bipm.org/utils/common/pdf/monographieRI/Monographie_BIPM-5_Tables_Vol1.pdf
http://www.bipm.org/utils/common/pdf/monographieRI/Monographie_BIPM-5_Tables_Vol2.pdf
http://www1.bipm.org/utils/common/pdf/monographieRI/Monographie_BIPM-5_Tables_Vol3.pdf
http://www.bipm.org/utils/common/pdf/monographieRI/Monographie_BIPM-5_Tables_Vol4.pdf
updated at: http://www.nucleide.org/DDEP_WG/DDEPdata.htm.

Exception: The ^{230}Th and ^{243}Cm half-lives are based on the *Evaluated Nuclear Structure Data File (ENSDF), online database,* Y.A. Akovali, *Nuclear Data Sheets* **58**, 555 (1989), National Nuclear Center, Brookhaven National Laboratory (Upton, NY), accessed February 2010.
Refer to http://www.nndc.bnl.gov/ensdf/

(4) The laboratory value represents the mean of five replicate measurements. The reported relative expanded uncertainty includes a coverage factor of **k =2**.

(5) The **Difference** quoted is the difference between the **NIST Value,** V_N, and the **Reported Value**, V_R, expressed as a percent relative to the **NIST Value**, $\{(V_R - V_N)/V_N \cdot 100\%\}$.

(6) Test samples were prepared by gravimetric dilutions of NIST calibrated solutions and SRM's. These solutions and SRM's were calibrated using the following activity measurement methodologies:

	Nuclide	Methodology and measuring instrument(s)
a)	^{57}Co	NIST 4πγ calibrated ionization chamber
b)	^{60}Co	Pressurized "4π"γ ionization chamber calibrated using a cobalt-60 solution whose activity was determined by 4πβ - γ coincidence and anticoincidence counting.
c)	^{90}Sr	4πβ liquid-scintillation counting. The Sr-90 plus Y-90 detection efficiency was calculated using the CIEMAT/NIST method with H-3 as the detection-efficiency monitor.
d)	^{137}Cs	Pressurized "4π"γ ionization chamber calibrated using a cesium-137 solution whose activity was determined by 4π(e + X) - γ anticoincidence counting. Pressurized "4π"γ ionization chamber calibrated using a cesium-137 solution whose number of cesium-137 atoms was determined by isotope-dilution mass spectrometry.
e)	^{210}Pb	Three 4παβ liquid-scintillation counting systems. The Pb-210 plus Bi-210 plus Po-210 detection efficiency was calculated using the CN2003 code for the CIEMAT/NIST method with composition matched H-3 as the detection-efficiency monitor.
f)	^{226}Ra	NIST pressurized "4π"γ ionization chamber calibrated with the national radium standards.
g)	^{230}Th	Two 4πα liquid scintillation counting systems
h)	^{234}U	Mass spectrometry, silicon surface barrier alpha-spectrometry, and 4παβ liquid-scintillation counting systems
i)	^{235}U	(same as for U-234)
j)	^{238}U	(same as for U-234)
k)	^{238}Pu	Two 4πα liquid scintillation counting systems
l)	^{240}Pu	NIST 0.1π alpha defined solid angle counter with scintillation detector, two 4πα liquid scintillation counting systems, and a silicon surface barrier α-spectrometry system
m)	^{241}Am	NIST 4πα liquid-scintillation counting system
n)	^{243}Cm	NIST 0.1π and 0.8π alpha defined solid angle counters with scintillation detectors

(7) Summary of the Reporting-Laboratory methodologies.

(8) ANSI N42.22 defines the acceptance criteria for verification testing by NIST as:

$$|V_R - V_N| < 3 * \sqrt{u_{c,R}^2 + u_{c,N}^2}$$

Where: V_N = NIST Value;
V_R = Reported Value;
$u_{c,N}$ = Standard combined uncertainty of the NIST value, V_N;
$u_{c,R}$ = Standard combined uncertainty of the Reported value, V_R; and
$3 * \sqrt{u_{c,R}^2 + u_{c,N}^2}$ = Traceability Limit (limit to which measurement traceability may be claimed with 99% confidence)

(9) ANSI N13.30 defines the criteria for acceptable bias between -25 and +50 percent, and acceptable precision between -40 and +40 percent, 1 sigma total propagated uncertainty.

Reference:

ANSI National Standards Institute, ANSI N42.22-1995, "Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control"

ANSI National Standards Institute, ANSI N13.30-1996, "Performance Criteria for Radiobioassay"

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