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

RESULTS OF THE EXCRETA BIOASSAY QUALITY CONTROL PROGRAM FOR APRIL 1, 2010 THROUGH MARCH 31, 2011

CL Antonio

July 2012

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Pacific Northwest National Laboratory
Richland, Washington 99352

RESULTS OF THE EXCRETA BIOASSAY QUALITY CONTROL PROGRAM FOR APRIL 1, 2010 THROUGH MARCH 31, 2011 CONTRACT 112512

Cheryl L. Antonio

April 2012

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

SUMMARY

A total of 76 urine samples and 10 spiked fecal samples were submitted during the report period (April 1, 2010 through March 31, 2011) to GEL Laboratories, LLC in South Carolina by the Hanford Internal Dosimetry Program (IDP) to check the accuracy, precision, and detection levels of their analyses. Urine analyses for ^{14}C, Sr, for ^{238}Pu, ^{239}Pu, ^{241}Am, ^{243}Am, ^{235}U, ^{238}U, ^{238}U-mass and fecal analyses for ^{241}Am, ^{238}Pu and ^{239}Pu were tested this year. The number of QC urine samples submitted during the report period represented 1.1% 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 31% of the analyses processed by GEL during the first year of contract 112512 were quality control samples. GEL tested the performance of 23 radioisotopes, all of which met or exceeded the specifications in the Statement of Work within statistical uncertainty except the slightly elevated relative bias for $^{243,244}Cm$ (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 Fifth 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 within statistical variation. Of the 89 samples that GEL spiked at the CDL, only two samples did not show detection, giving a false-negative (beta error) of 2%, which was acceptable.

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, $^{233,234}U$ was not evaluated. However, 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 ^{238}U mass urinalysis program using inductively-coupled plasma mass spectrometry (ICPMS) and it was considered acceptable. Because IDP uses a 0.2 μg screening level for ^{238}U mass, 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 as tested by IDP met the acceptance criteria. The bias and precision was tested by IDP at 0.2 μg and by GEL at 1 μg/sample and at 0.05 μg/sample.

The performance statistics for the ^{236}U analysis using ICPMS were supplied by GEL's QC program, IDP did not submit QC samples. The MDA and relative bias and precision reported by GEL met the performance criteria. The ^{236}U analysis procedure was considered acceptable.

The total strontium procedure is used to screen samples to determine whether analysis for ^{90}Sr is warranted. 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 ingrowth 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 46% of the CL. The MDA, relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all within limits. The relative bias was slightly elevated but within statistical uncertainty. The 16 samples spiked at the contractual level by IDP were all detected. The strontium urinalysis procedure was concluded to be acceptable.

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 MDA tested by IDP was slightly above the CDL, but the difference was not considered significant because only three results were submitted. The MDA tested by GEL and based on 557 samples was 25% less than the criteria. The 14 samples spiked at the CL for ^{239}Pu all showed detection and the relative bias and precision met the acceptance criteria. Out of 555 samples spiked by GEL at the CDL, only 2 samples did show detection, giving a false-negative (beta error) of 0.4%, which was acceptable. There were 17 blank samples analyzed for ^{238}Pu activity, none of the 17 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 likewise acceptable. More than 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 with a variation of less than 3 sigma. The fecal aliquoting procedure was acceptable. This year IDP submitted 10 actual fecal samples, five samples were blanks and five samples were spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The MDA, precision and bias for ^{239}Pu and ^{238}Pu met the performance criteria. The performance statistics reported by GEL for ^{239}Pu and ^{238}Pu also met the acceptance criterion. The failed analysis rate for fecal sampling was 1% with a low or high yield rate of 7%, which is within the contractual level of 10%. Overall the plutonium fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

The ^{241}Am fecal and urine analyses met the acceptance criteria for MDA, relative bias and precision. The MDA tested by IDP was slightly above the CDL, but the difference was not considered significant because only three results were submitted. The MDA as reported by GEL was less than 10% of the contractual level. All 14 of the ^{241}Am samples spiked at the contractual detection level (CDL) were detected. Out of 280 samples spiked by GEL at the CDL, only three samples did not show detection, giving a false-negative (beta error) of 1%, which was acceptable. 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 with a variation less than three sigma. This year IDP submitted five

actual fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. Overall the ^{241}Am fecal analyses were considered acceptable.

The AM243 procedure was identical to the AM241 procedure, except a different tracer is used (^{244}Cm instead of ^{243}Am). The four blank ^{243}Am QC samples submitted were all reported with results less than the decision level, and the calculated MDA met the contractual detection level. The performance statistics for ^{243}Am, as tested by GEL, met the acceptance criteria for relative bias and precision. The MDA was slightly elevated, but was within the statistical uncertainty of the analysis. 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 average relative bias of ^{244}Cm was slightly elevated but it was not considered a concern (see Table 4). Overall 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. Of the 12 samples spiked with ^{232}Th, one sample did not show detection, resulting in a false-negative percent (beta error) of 8%, which was determined to be acceptable, assuming the normal statistical variation in the measurement process. Overall the results met the acceptance criteria and the isotopic thorium urinalysis program was considered acceptable.

Neptunium-237 was likewise not tested by IDP and the performance statistics were supplied by the GEL's QC program. The MDA, average relative bias and precision met the performance criteria and the NP237 analysis was considered acceptable.

Because GEL LLC, did not meet the acceptance criteria for C14 urinalyses under the guidelines set forth by the Department of Energy's Laboratory Accreditation Program (DOELAP), IDP submitted 12 samples for analysis to further test the procedure. Five blank samples and 12 spiked samples were submitted and the MDA, average relative bias and precision all met IDP's contractual specifications. After reviewing the ^{14}C source material on the Hanford Site, IDP determined that ^{14}C bioassay monitoring would not be required and pursuing DOELAP accreditation for the procedure was discontinued. This is discussed further in the Carbon-14 section.

CONTENTS

TABLES

INTRODUCTION

This report summarizes the results of the excreta bioassay quality control program's monitoring of the performance of GEL Laboratories, LLC (GEL) for samples submitted from April 1, 2010 through March 31, 2011 under contract 112512. During the reporting period GEL analyzed, under the contract with Battelle, 7455 urine and 96 fecal samples for various radionuclides. The number of samples analyzed was much greater than in previous years due to an increased work force due in part to the American Recovery Act.

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 2010).

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 2010) 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, ^{14}C, ^{237}Np, ^{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 (alpha spectrometry and mass analysis) and fecal samples for ^{14}C, ^{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 112512 – Feb. 2010

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[a] Email	Electronic	Written	Email Reporting Limit; (dpm/sample) [b] 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[c]	0.02		20				Eq. 1	
U(∝) Isotopic (IU)	U-234, U-235, U-238	0.02		20				(d)	
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	10 dpm/ml		5				10 dpm/ml	
Sr-total (SR)	Sr (sum Sr-89 + Sr-90)	10		20				5	
Sr-90 (SR90)[e]	Sr-90	10		30				5	
Gamma Spectroscopy (ISPEC)	K-40, Cs-137 + Others[f]	See Table B-5		20				Eq. 1	
Gamma Spectroscopy (LEPD)	Am-241	5		20				Eq. 1	
U-236 Mass (U 236)	U-236	0.000140 µg/sample[g]		20				70 pg/sample	
U-238 Mass (U 238)	U-238	0.06 µg/sample	0.3	20				0.2 µg/sample	
Pm-147 (PM147)	Pm-147	50	200	20				Eq. 1	
Sequential Analyses:									
Pu(∝) Iso and Sr-total (IPS)	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-238 (IUPU)				25					
Actinide(∝) Isotopic (ITPAC)[h]				25					
Cm(∝) Iso, Am-241 (ICA)	Cm-242, Cm-244, Am-241[c]			20					
Pu(∝) Iso and U ISO (IPIU)				25					

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

(b) Email report required only when analytical results exceed level specified.

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

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

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

(f) Report all isotopes present at levels exceeding Equation 1. 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.

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

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

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 (∝) 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.
(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	FIFTH CONTRACT (11530) YEAR - GEL 4/1/09 through 3/31/10				FIRST CONTRACT (112512) YEAR - GEL 4/1/10 through 3/31/11			
Code[a]	Total	IDP QC	%	GEL QC[b]	Total	IDP QC	%	GEL QC[b]
Urine								
H3	1285	0	--	388	234	0	--	148
SR90, SR	406	0	--	882	293	2	1	653
C14	--	0	--	--	12	12	100%	--
AM241	158	0	--	701	317	0	--	842
AM243	103	6	6%	108	23	4	17%	42
U235	--	0	--	--	--	0	--	--
ICM	25	0	--		67	0	--	208
IPU	1730	2	0.1%	1742	1423	0	--	1669
IPUL	--	0	--	--	--	0	--	--
IPA	596	0	--	N/A	1232	0	--	N/A
IPS	925	0	--	N/A	996	0	--	N/A
IPSA	323	23	7%	N/A	239	17	7%	N/A
IPSR	--	0	--	--	--	0	--	--
ISPEC	--	0	--	--	2	0	--	--
ITPAC	271	0	--	N/A	180	0	--	N/A
ITH	21	0	--	48	15	0	--	36
IUPU	127	0	--	N/A	178	0	--	N/A
IPIU	38	0	--	N/A	26	0	--	N/A
IU	726	10	1%	465	410	12	3%	267
NP237	13	0	--	24	7	0	--	15
U236	9	0	--	17	9	0	--	24
U238 mass	1314	17	1%	709	1792	29	2%	28
LEPD	--	0	--	--	--	0	--	--
PU241	--	0	--	--	--	0	--	--
Total	*8070*	*58*	*1%*	*5084*	*7455*	*76*	*1%*	*3932*
Fecal [c]								
ICM	--	0	--	--	4	0	--	6
AM241	2	0	--	88	2	0	--	133
IPU	1	0	--	88	1	0	--	126
IPA	57	10	18%	N/A	89	10	11%	N/A
Total	*60*	*10*	*17%*	*176*	*96*	*10*	*10%*	*265*

[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 76 urine and 10 fecal QC samples submitted by IDP to GEL during the reporting period, GEL reported 7455 analytical urine results for 18 different analytes and 96 fecal results for 5 different analytes. The 86 QC samples represent 1.1% of the total analyses performed by GEL. In addition to these samples, GEL analyzed 4764 internal QC samples. The QC samples analyzed equaled 31% 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, implemented on April 1, 2005 with GEL, specifies an operational year that ends March 31st, each year. This QC report covers the fourth operational year of that contract, and includes samples analyzed by GEL during period of April 1, 2008 through March 31, 2009.

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.

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 2009-2010	Counter Efficiency 2010-2011	Chemical Yield 2009-2010	Chemical Yield 2010-2011
Urine	^{3}H	20	*20*	0.243	0.243	N/A	N/A
	Total Sr	45	*10*	0.379	0.379	0.778	0.707
	SR90	45	*10*	0.379	0.379	0.759	0.707
	^{241}Am	2520	*0.02*	0.391	0.391	0.867	0.869
	^{243}Am	2520	*0.02*	0.391	0.391	0.922	0.867
	$^{242}Cm/^{244}Cm$	2520	*0.02*	0.391	0.391	0.867	0.869
	^{237}Np	2520	*0.02*	0.391	0.391	0.717	0.648
	$^{239}Pu/^{238}Pu$	2520	*0.02*	0.391	0.391	0.902	0.74
	IPUL	10000	*0.005*	---	---	---	---
	$^{228}Th/^{230}Th/^{232}Th$	2520	*0.1*	0.386	0.386	0.900	0.765
	$^{234}U/^{235}U/^{238}U$	2520	*0.02*	0.386	0.386	0.862	0.87
	^{238}U mass	--	*0.06*	N/A	N/A	N/A	N/A
Fecal	^{241}Am	960	*0.8*	0.391	0.391	8.865	0.864
	$^{238}Pu/^{239}Pu$	960	*0.2*	0.391	0.391	0.801	0.827

(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.

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

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 22 urines and 7 artificial fecal samples prepared by RPG. 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). In future QC reports GEL has been asked not to classify QC data points as outliers and remove them from the database if the result was a statistical anomaly. However, if there was a laboratory error resulting in an erroneous result, then the associated data can be excluded from the performance statistics. Any outliers removed from the data tables need to be addressed in the observation section.

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	74	0.5050	0.808	**20**	74	-0.01	0.08	0	…	…
^{14}C (dpm/ml)	**IDP**	5	0.304	0.800	**10**	0	…	…	7	-0.091	0.063
Total Sr/^{90}Sr	**IDP**	3	1.707	4.119	**10**	16	-0.053	0.09	0	…	…
	GEL	218	0.73	4.65	**10**	217	0.12 (e)	0.18	218	0.03	0.09
^{237}Np	GEL	5	0.00	0.00	**10**	5	0.19	0.30	5	0.018	0.092
^{228}Th	GEL	12	0.054	0.071	**0.1**	0	…	…	0	…	…
^{229}Th	GEL	12	0.026	0.038	**0.1**	0	…	…	0	…	…
^{232}Th	GEL	12	0.020	0.031	**0.1**	12	-0.03	0.31	12	-0.006	0.07
^{230}Th	GEL	12	0.035	0.051	**0.1**	0	…	…	0	…	…
^{242}Cm	GEL	71	0.004	0.009	**0.02**	0	…	…	0	…	…
^{243,244}Cm	GEL	71	0.004	0.010	**0.02**	69	0.14 (c)	0.27	68	-0.095	0.019
^{238}Pu-urine	**IDP**	17	0.003	0.011	**0.02**	0	…	…	0	…	…
	GEL	557	0.005	0.013	**0.02**	0	…	…	0	…	…
feces	**IDP**	5	0.011	0.037	**0.2**	0	…	…	5	-0.079	0.080
	GEL	42	0.02	0.060	**0.2**	0	…	…	0	…	…
^{239,240}Pu-urine	**IDP**	3	0.006	0.023 (e)	**0.02**	14	0.046	0.294	0	…	…
	GEL	557	0.008	0.017	**0.02**	555	0.04	0.30	557	0.01	0.06
feces	**IDP**	5	0.011	0.036	**0.2**	0	…	…	5	-0.155	0.177
	GEL	42	0.04	0.096	**0.2**	42	-0.01	0.21	42	0.004	0.064
^{241}Am-urine	**IDP**	3	0.008	0.025 (e)	**0.02**	14	0.00	0.18			
	GEL	281	0.009	0.018	**0.02**	280	0.015	0.29	281	-0.086	0.081
feces	**IDP**	5	0.032	0.079	**0.2**	0	…	…	5	-0.123	0.111
	GEL	43	0.04	0.105	**0.2**	43	0.04	0.21	42	-0.048	0.073
^{243}Am-urine	**IDP**	4	0.006	0.019	**0.02**	0	…	…	0	…	…
	GEL	14	0.011	0.021	**0.02**	14	0.09	0.57	14	0.01	0.10
^{233,234}U	**IDP**	0	…	…	**0.02**	0	…	…	0	…	…
	GEL	89	0.018	0.031	**0.02**	0	…	…	0	…	…
^{235,236}U	**IDP**	12	0.00	0.011	**0.02**	0	…	…	0	…	…
	GEL	89	0.009	0.018	**0.02**	0	…	…	0	…	…
^{238}U	**IDP**	0	…	…	**0.02**	0	…	…	12	0.043	0.108
	GEL	89	0.017	0.030	**0.02**	89	0.06	0.33	89	0.03	0.11
^{236}U (ICPMS)[(b)]	**IDP**	0	…	…	**140 pg**	0	…	…	0		
	GEL	8	1.258	36.408	**140 pg**	8	-0.11	0.34	8	-0.03	0.05
^{238}U (ICPMS) [(b)]	**IDP**	2	0.005	0.051	**0.06 µg**	0	…	…	27	-0.05	0.24
	GEL	150	0.006	0.02	**0.06 µg**	150	0.09	0.18	150	-0.009	0.064

(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	2010	0	…	…	0	…	…	0	…	…
		2009	0	…	…	0	…	…	0	…	…
Sr	10 dpm	2010	3	1.7	4.1	16	-0.05	0.09	0	…	…
		2009	0	…	…	23	-0.05	0.12	0	…	…
U	0.06 mg	2010	2	0.01	0.05	0	…	…	27	-0.05	0.24
(ICPMS)		2009	0	…	…	0	…	…	17	-0.02	0.32
^{235}U	0.02 dpm	2010	12	0.003	0.011	0	…	…	0	…	…
		2009	10	0.004	0.012	0	…	…	0	…	…
^{238}U	0.02 dpm	2010	0	…	…	0	…	…	12	0.04	0.11
		2009	0	…	…	10	0.02	0.09	0	…	…
^{238}Pu	0.02 dpm	2010	17	0.00	0.01	0	…	…	0	…	…
(urine)		2009	25	0.00	0.01	0	…	…	0	…	…
^{238}Pu	0.2 dpm	2010	5	0.01	0.04	0	…	…	5	-0.08	0.08
(fecal)		2009	5	0.01	0.04	5	0.08	0.19	0	…	…
^{239}Pu	0.02 dpm	2010	3	0.006	0.023 (e)	14	0.05	0.29	0	…	…
(urine)		2009	5	0.011	0.036	5	0.08	0.19	0	…	…
^{239}Pu	0.2 dpm	2010	5	0.01	0.04	0	…	…	5	-0.16	0.18
(fecal)		2009	5	0.01	0.04	5	-0.02	0.13	0	…	…
^{241}Am	0.02 dpm	2010	3	0.008	0.025 (e)	14	0.00	0.18	0	0.00	0.00
(urine)		2009	0	…	…	18	-0.06	0.21	5	0.104	0.209
^{241}Am	0.2 dpm	2010	5	0.03	0.08	0	…	…	5	-0.12	0.11
(fecal)		2009	0	…	…	6.0	-0.03	0.06	0	…	…
^{243}Am	0.02 dpm	2010	4	0.006	0.019	0	…	…	0	…	…
		2009	6	0.004	0.013	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 2010-2011 Yield Flags	Analytical Samples 2010-2011 Failed Analyses
Urine								
^{3}H	0	0 (0)	0	74		0 (0)		
Sr	19	0 (0)	16	217	0 (0)	0 (0)	5%	1%
^{235}U	12	0 (0)	0	0			5%	4%
^{238}U[(a)]	12	0 (0)	12	89	0 (0)	2 (2%)		
^{238}Pu	17	0 (0)	0	0			7%	1%
^{239}Pu	17	0 (0)	14	555	0 (0)	2 (0.4%)	7%	1%
^{241}Am	17	0 (0)	14	280	0 (0)	3 (1%)	1%	1%
^{243}Am	4	0 (0)	0	14				
U-ICPMS [(a)]	29	0 (0)	27	37	0 (0)	0 (0)		
Total	*127*		*83*	*1266*				
Feces								
^{241}Am	10	0 (0)	5 [(a)]	43	0 (0)	0 (0)	2%	1%
^{238}Pu	10	0 (0)	5 [(a)]	0	0 (0)	0 (0)	7%	1%
^{239}Pu	10	0 (0)	5 [(a)]	42	0 (0)	0 (0)	7%	1%
Total	*30*		*15*	*85*				

(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 whether analysis for ^{90}Sr is warranted. 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 ingrowth 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 46% of the CL. The MDA, relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all limits. The relative bias was slightly elevated but within statistical uncertainty. The 16 samples spiked at the contractual level by IDP were all detected. The strontium urinalysis procedure was concluded to be acceptable.

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 MDA tested by IDP was slightly elevated but only three samples were submitted and the statistical variation was therefore high. The MDA tested by GEL and based on 557 samples was 25% less than the criteria. The 14 samples spiked at the CL for ^{239}Pu all showed detection and the relative bias and precision met the acceptance criteria. Out of 555 samples spiked by GEL at the CDL, only 2 samples did show detection, giving a false-negative (beta error) of 0.4%, which was acceptable. There were 17 blank samples analyzed for ^{238}Pu activity, none of the 17 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 likewise acceptable. More than 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 with a variation of less than 3-sigma . The fecal aliquoting procedure was acceptable. This year IDP submitted 10 actual fecal samples, five samples were blanks and five samples were spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The MDA, precision and bias for ^{239}Pu and ^{238}Pu met the performance criteria. The performance statistics reported by GEL for ^{239}Pu and ^{238}Pu also met the acceptance criterion. The failed analysis rate for fecal sampling was 1% with a low or high yield rate of 7%, which is within the contractual level of 10%. Overall the plutonium fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

ISOTOPIC URANIUM

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 within statistical variation. Of the 89 samples that GEL spiked at the CDL, only two samples did show detection, giving a false-negative (beta error) of 2%, which was acceptable.

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, $^{233,234}U$ was not evaluated. However, 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 ^{238}U mass urinalysis program using inductively-coupled plasma mass spectrometry (ICPMS) and it was considered acceptable. Because IDP uses a 0.2 µg screening level for ^{238}U mass, 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 first quarter GEL recorded ^{238}U mass results as Total Uranium, a convention used when the KPA system was used for total uranium mass analyses. This was in error because the results were not total uranium but ^{238}U mass by ICPMS, the error was identified before the end of the first quarter and the problem was corrected. However, the relative bias and precision were reported separating for the quality control samples labeled as Total Uranium results and ^{238}U mass (ICPMS). The values listed in Table 4 for relative bias and precision are a compilation of the two datasets. The relative bias and precision were likewise acceptable. The bias and

precision as tested by IDP met the acceptance criteria. The bias and precision were tested by IDP at 0.2 µg and by GEL at 1 µg/sample and at 0.05 µg/sample.

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

The performance statistics for the ^{236}U analysis using ICPMS were supplied by GEL's QC program, IDP did not submit QC samples. 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 analyses met the acceptance criteria for MDA, relative bias and precision. The MDA tested by IDP was slightly elevated but only three samples were submitted and the statistical variation was therefore high. The MDA as reported by GEL was less than 10% of the contractual level. All 14 of the ^{241}Am samples spiked at the contractual detection level (CDL) were detected. Out of 280 samples spiked by GEL at the CDL, only three samples did show detection, giving a false-negative (beta error) of 1%, which was acceptable. 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 five actual fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. Overall the ^{241}Am fecal analyses were considered acceptable.

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 50% of the contractual detection level. The performance statistics for ^{243}Am, as tested by GEL, met the acceptance criteria. The AM243 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 average relative bias of ^{244}Cm was slightly elevated but it was not considered a concern (see Table 4). Overall 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. Of the 12 samples spiked with ^{232}Th, one sample did not show detection, resulting in a false-negative percent (beta error) of 8%, which was determined to be acceptable, assuming the normal statistical variation in the measurement process. Overall the results met the acceptance criteria and the isotopic thorium urinalysis program was considered acceptable.

NEPTUNIUM-237

Neptunium-237 was likewise not tested by IDP and the performance statistics were supplied by the GEL's QC program. The MDA, average relative bias and precision met the performance criteria and the NP237 analysis was considered acceptable.

CARBON-14

Anticipating that decommissioning and decontamination (D&D) work in the old production reactors on the Hanford site might begin in the next few years, IDP requested the Department of Energy Laboratory Accreditation Program (DOELAP) certification for routine ^{14}C urinalyses. The current statement of work only specified non-routine ^{14}C bioassays, which was outside the scope of DOELAP. DOELAP Test Session 13, included ^{14}C urine samples for GEL to analyze and report. However, GEL LLC did not meet the acceptance criteria for ^{14}C urinalyses under the guidelines set forth by DOELAP. GEL's analytical results showed biases ranging from -0.51 to -0.65, the DOELAP acceptable range was -0.25 to 0.5. As follow-up, IDP submitted 12 samples for analysis to further test the procedure. Five blank samples and 12 spiked samples were submitted and the MDA, average relative bias and precision all met IDP's contractual specifications. GEL requested from DOELAP another set of samples and once again GEL's analytical results showed a negative bias that did not meet the acceptance criteria. A review of the DOELAP prepared samples identified differences in the sample preparation and spike material that would require additional handling than covered in GEL's routine procedures.

Before proposing a re-evaluation of the ^{14}C analysis procedure, IDP reviewed the criteria for a ^{14}C bioassay routine monitoring program. Based on the source material, characterization data and proposed D&D work, a routine ^{14}C bioassay monitoring program would not be needed. The request for DOELAP certification for ^{14}C urinalyses was withdrawn and additional performance evaluation samples were not requested. Appendix A includes the documentation reviewing the need for a ^{14}C bioassay monitoring program at the Hanford site and email correspondences addressing the DOELAP samples and IDP ^{14}C audit samples.

FOLLOW-UP ON CONCERNS DURING THE FIFTH CONTRACT YEAR

There were a few concerns carried over from the fourth contract year, primarily technician errors involving sample batches, typically consisting of loss of sample (e.g, dropping, breakage and spillage), cross contamination, forgetting to perform a task, or lack of proper documentation. Over the year there were about 5 separate incidents resulting in 38 failed analyses including plutonium, strontium and isotopic uranium. The failure rate was still well below 10% for all analytes.

Incident reports issued during the fourth contract year and their follow-up are reported in Appendix B.

SUMMARY OF THE BIOASSAY QUALITY CONTROL REPORT FROM GEL INCORPORATED, FOR THE CONTRACT 112512 FIRST YEAR 2010/2011[(a)]

GEL reported all analytical batches were analyzed with a reagent blank (Umass only), matrix blank or both. GEL considered blanks in control when the calculated 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. The urine sampling program showed acceptable levels for low-yields for all analyses. The isotopic plutonium urinalysis and fecal analysis program showed the highest low yield rate at 7%, which is below the 10% level for follow-up.

RESULTS FROM INTERCOMPARISON PROGRAMS

GEL participated in two intercomparison programs (Appendix C – Intercomparison Programs) in the first contract year. Between August and October 2010, GEL participated in the National Institute of Standards and Technology's program testing the relative bias and precision for ^{60}Co, ^{137}Cs, ^{238}Pu, $^{240,239}Pu$, ^{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. 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, ^{226}Ra, ^{238}Pu, $^{240,239}Pu$, ^{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.

In 2010 GEL participated in session 13 of DOELAP and was tested for ^{60}Co, ^{137}Cs, ^{238}Pu, $^{240,239}Pu$, ^{241}Am, ^{230}Th, ^{228}Th, ^{232}Th, ^{237}Np, ^{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 in feces. For the urine program, GEL was tested in ^{14}C, ^{3}H, ^{60}Co, ^{137}Cs, ^{238}Pu, $^{240,239}Pu$, ^{241}Am, ^{230}Th, ^{228}Th, ^{232}Th, ^{237}Np, ^{235}U, ^{238}U, ^{234}U, ^{238}U-mass and ^{90}Sr in synthetic urine. GEL passed the performance statistics for relative bias and precision for all isotopes except ^{14}C, which was discussed above in the CARBON-14 section.

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. 2010. "Contract No. 112512 Bioassay Radiochemical Analytical Services" General Engineering Laboratories, LLC, Charleston, South Carolina. (Available from the Hanford Radiological Records Historical File.)

(a) Summaries are taken from GEL (2010).

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.

Pizarro, S. 2009. " PNNL Annual QC Package, Fifth 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.)

Pizarro, S. 2010. " PNNL Annual QC Package, First Contract Year 2010/2011 (April 1, 2010 – March 31, 2011)" 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.. 2012. "Results of the PNL Excreta Bioassay Quality Control Oversight Program for April 1, 2010 through June 30, 2011." Letter Report, Pacific Northwest Laboratory, Richland, Washington. (Available from the Hanford Radiological Records Historical File.)

Antonio, C. L. 2012. "Results of the PNL Excreta Bioassay Quality Control Oversight Program for July 1, 2010 through September 30, 2010." Letter Report, Pacific Northwest Laboratory, Richland, Washington. (Available from the Hanford Radiological Records Historical File.)

Antonio, C. L. 2012. "Results of the PNL Excreta Bioassay Quality Control Oversight Program for October 1, 2010 through December 31, 2010." Letter Report, Pacific Northwest Laboratory, Richland, Washington. (Available from the Hanford Radiological Records Historical File.)

ATTACHMENT 1 – CARBON-14 FOLLOW UP

Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Thursday, April 19, 2012 3:33 PM
To: Antonio, Cheryl L
Subject: C-14 reports

Attached are the PNNL report and the ORS journal publication. Simply put, based on the conclusions of these reports and the available data for graphite reactor decommissioning, there does not appear to be a likely need for any kind of routine C-14 bioassay. Hence the effort to achieve accreditation in the C-14 category is not being pressed by PNNL.


PNNL-SA-75300 C-14 Bioassay fu...


Carbon_14_Bioassay_for_Decommi...

Gene

Eugene H. Carbaugh, CHP
Staff Scientist and Internal Dosimetry Manager
Pacific Northwest National Laboratory
Phone: (509) 376-6632

U.S. DEPARTMENT OF ENERGY

PNNL-SA-75300

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

Carbon-14 Bioassay for Decommissioning of Hanford Reactors

EH Carbaugh
DJ Watson

September 2010

Pacific Northwest
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PNNL-SA-75300

Carbon-14 Bioassay for Decommissioning of Hanford Reactors

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Prepared for
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Pacific Northwest National Laboratory
Richland, Washington 99352

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CARBON-14 BIOASSAY FOR DECOMMISSIONING OF HANFORD REACTORS

1.0 Introduction

The old production reactors at the US Department of Energy Hanford Site used large graphite blocks as the moderator. The last of these reactors was permanently shut down in 1986, and the long range site plan for their decommissioning has been to cocoon them for 75 years to allow the relatively short half-life radionuclides to decay away. After 75 years the intent was to transport the graphite piles several miles to a below ground disposal facility. Funds being made available under the American Recovery and Reinvestment Act of 2009 may result in some or all of this work occurring much sooner than was originally planned. Questions have been raised about the potential need for 14C bioassay of workers who might be involved in this work, and the technical issues associated with such bioassay.

Carbon-14 decays with a half-life of 5730 years by pure beta decay. The beta particle emitted has a low average energy of 49.45 keV. It is formed naturally in the upper atmosphere by cosmic ray interactions with 14N. The natural abundance of 14C is approximately one part per trillion (10-12), or about 6 pCi/gC (NCRP 1985).

2.0 The Hanford Reactors as a Source of ^{14}C

Carbon-14 is an unavoidable activation product in nuclear reactors such as the former Hanford production reactors that used graphite moderated cores. Carbon-14 production arises from neutron bombardment through one of two processes: the 14N (n, p)14C reaction, with a capture cross section of 1.8 barns, and the 13C(n, γ)14C transmutation, with a capture cross section of 0.0009 barns. Nitrogen impurities found in the graphite, the water coolant and the core cover gas all contribute to 14C production. Thus, carbon-14 concentrations can fluctuate between reactors due to varying levels of these impurities and reactor operating conditions. The nitrogen source also affects the form of the resulting 14C: nitrogen impurities in graphite lead to 14C incorporated in the graphite lattice or pore spaces, while coolant or cover gas nitrogen leads to carbonaceous solids or amorphous carbon deposited on the surfaces and open pore spaces of the graphite (EPRI 2006).

Hanford reactors contain between 1400 and 2800 tons of graphite per reactor. Graphite samples in 1958, 1967, 1976, 1977 and the early 1980s showed 14C concentrations ranging from 0.2 to 3.4 µCi/g (Paasch 1985, Miller and Steffes 1987), with a nominal mean of 2.4 µCi/g. Samples collected included powder from a reactor process core tube broaching tool (likely representing 14C generated primarily from neutron reactions with the nitrogen cover gas) and core drillings (likely representing 14C created from nitrogen impurities in the graphite matrix and activation of 13C).

3.0 Behavior of 14C in the Human Body

Reference Man (ICRP 1974) shows the carbon content for an adult male to be 16,000 g with the equilibrium carbon balance being 300 g/d intake in food and fluids, and losses of 270 g/d by exhalation, 5 g/d by urine (mostly as urea), 7 g/d by feces, and for the remaining 18 g/d through all other losses (e.g., sweat). Using these parameters, and the environmental abundance of 14C as 10-12, the Reference Man 14C body content is about 71 nCi. Based on the carbon balance, background excretion of 14C is estimated at 30 pCi/d in urine and 42 pCi/d in feces. Assuming 1600 mL/d urine excretion and 150 g/d

fecal excretion of Reference Man (ICRP 2002), the respective concentrations would be about 0.04 dpm/mL and 0.6 dpm/g.

Absorption of 14C in the respiratory tract is highly dependent on its chemical form, as described in ICRP 68 (1994) and ICRP 71 (1995) for gases or vapors and ICRP 71 for particulates. Carbon monoxide (14CO) is considered Vapor Class SR-1, a soluble or reactive gas or vapor, with 40% of the inhalation instantaneously absorbed to blood and bound to hemoglobin and 60% exhaled. As carbon dioxide (14CO2) or an organic compound, it is described as Vapor Class SR-2, highly soluble or reactive gas or vapor, with 100% of the inhalation completely and instantaneously absorbed into blood from the respiratory tract. The biological half-time used for inhaled carbon monoxide absorbed in the body is 200 minutes. Inhaled carbon dioxide is assumed to be uniformly distributed throughout all organs and tissues.

Carbon can exist as all three particulate absorption types, Type F, M, or S. Rat studies cited in ICRP 71 indicated diesel exhaust particles exhibited Type M behavior, whereas 14C-bearing material obtained from air filters during re-tubing of a CANDU reactor were consistent with Type S material. Graphite is a highly insoluble crystalline form of carbon that is considered to demonstrate Type S behavior. Any 14C formed within the graphite matrix is likewise considered to demonstrate Type S behavior. The f1 absorption fractions for Types F, M, and S are respectively, 1, 0.1 and 0.01. Type F carbon compounds are not specifically identified in the ICRP publications, however are considered to be particulate aerosols of organic compounds.

Once absorbed in the blood, the biokinetic model for metabolized carbon in the body assumes a 100% distribution uniformly in all organs and tissues from which it clears with a 40-day biological half-time.

4.0 Internal Dosimetry Factors

The ICRP has compiled committed effective dose coefficients [e(50)] for 14C in the dioxide, monoxide, labeled methane, and organic gases and vapors forms in the ICRP CD-1 database (ICRP 2001). In addition, ICRP 71 tabulated effective dose coefficients for inhalation of absorption type F, M, and S particles, but using a particle size of 1-μm AMAD for public exposures instead of the 5-μm AMAD particle size recommended for occupational exposure. Thus, for this work of occupational monitoring program design, e(50) values assuming 5-μm AMAD particles were calculated using the IMBA Professional Plus™ computer code1, assuming the standard biokinetic models and a density of 2.16 g/cm3 for graphite particles. Results are shown in Table 1 along with the ICRP values for gases and vapors.

[1] IMBA Professional Plus is available from Health Protection Agency (HPA), Radiation Protection Division, Chilton, Didicot, Oxon., OX11 0RQ, UK, and from http://www.imbaprofessional.com/

Table 1. Effective dose coefficients, annual limits on intake and derived air concentrations for C-14 inhalations

Form of Inhalation Intake	Effective Dose Coefficient, *e*(50) (Sv/Bq)	Annual Limit on Intake (stochastic) (μCi)	Derived Air Concentration (μCi/mL)
^{14}C dioxide	6.50E-12[a]	2.1E+05[a]	7E-04[c]
^{14}C monoxide	8.00E-13[a]	1.7E+06[a]	8E-05[c]
^{14}C labeled methane	2.90E-12[a]	4.7E+05[a]	2.0E-04[a]
^{14}C organic gases and vapors	5.80E-12[a]	2.3E+03[a]	9E-07[c]
^{14}C Vapor Class SR-2	5.81E-12[b]	2.3E+03[b]	9.6E-07[b]
^{14}C Type F particulate	2.79E-10[b]	4.6E+02[b]	1.9E-06[b]
^{14}C Type M particulate	1.50E-09[b]	9.9E+02[b]	4.1E-07[b]
^{14}C Type S particulate	4.05E-09[b]	3.5E+02[b]	1.5E-07[b]

a.) based on ICRP CD-1 dose coefficients
b.) based on IMBA Professional Plus
c.) from 10 CFR 835 Appendix A (2007)

Table 1 also shows annual limits on intake (ALIs) and derived air concentrations (DACs). Stochastic ALIs were calculated for each of the exposure forms by dividing the United States regulatory limit of 5 rem effective dose by the e(50). DACs were then calculated for the type F, M, and S particulate intakes by dividing the ALI by the volume of air breathed by Reference Man in a year (2.4E+09 mL). Units were converted to the conventional units of the U.S. regulatory system. Also shown in Table 1 are the 14C DAC values contained 10 CFR 835 Appendix A (2007). Of particular note is that DACs for 14C particulates are two-to-three orders of magnitude smaller than the 14C DACs contained in 10 CFR 835 Appendix A. Monitoring programs which might utilize the most restrictive 10 CFR 835 DAC as a basis for workplace control, when in fact the source term would be a particulate instead of a gas or vapor, would seriously underestimate the significance of an intake based on air sampling data.

5.0 Bioassay Programs

Bioassay monitoring programs for 14C in workers typically rely on indirect bioassay using urine or fecal samples, with 14C converted to CO2, distillation, and then liquid scintillation counting.

Urine and fecal excretion fractions calculated using IMBA are shown in Table 2. For purposes of determining bioassay program design, intakes corresponding to 5-rem (the regulatory compliance level), 100-mrem (the investigation level), and 10-mrem (the recording level) were calculated using the e(50). These intakes were then multiplied by the respective urine and fecal excretion fractions to give derived reference bioassay levels at various times post intake, results for which are shown as side-by-side comparisons for urine and feces in Figure 1.

Table 2. Urine and fecal excretion fractions

Day Post Intake	Urine Excretion Fraction				Fecal Excretion Fraction			
	Type S[(a)]	Type M[(a)]	Type F[(a)]	SR-2	Type S[(a)]	Type M[(a)]	Type F[(a)]	SR-2
1	5.21E-07	9.31E-06	6.99E-05	1.68E-04	1.13E-01	1.00E-01	1.15E-05	3.01E-05
2	1.19E-06	1.84E-05	1.31E-04	2.76E-04	1.62E-01	1.39E-01	7.63E-05	1.73E-04
3	1.25E-06	1.91E-05	1.34E-04	2.78E-04	8.34E-02	7.09E-02	1.37E-04	2.94E-04
5	1.22E-06	1.86E-05	1.30E-04	2.69E-04	1.39E-02	1.18E-02	1.78E-04	3.70E-04
7	1.18E-06	1.82E-05	1.25E-04	2.60E-04	2.42E-03	2.05E-03	1.79E-04	3.71E-04
14	1.07E-06	1.67E-05	1.11E-04	2.30E-04	4.74E-04	3.89E-04	1.59E-04	3.31E-04
30	8.47E-07	1.36E-05	8.41E-05	1.75E-04	3.30E-04	2.55E-04	1.21E-04	2.51E-04
60	5.50E-07	9.30E-06	5.00E-05	1.04E-04	1.78E-04	1.22E-04	7.18E-05	1.49E-04
90	3.62E-07	6.42E-06	2.97E-05	6.17E-05	1.03E-04	6.39E-05	4.27E-05	8.87E-05
180	1.27E-07	2.38E-06	6.25E-06	1.30E-05	3.68E-05	1.59E-05	8.98E-06	1.86E-05
365	5.37E-08	5.51E-07	2.53E-07	5.26E-07	2.19E-05	3.80E-06	3.64E-07	7.55E-07
730	3.63E-08	6.18E-08	4.53E-10	9.42E-10	1.46E-05	4.25E-07	6.51E-10	1.35E-09
1825	1.52E-08	1.22E-10	2.60E-18	5.41E-18	4.61E-06	6.69E-10	3.74E-18	7.77E-18
3650	5.99E-09	6.39E-15	4.80E-32	9.96E-32	8.12E-07	2.04E-14	6.89E-32	1.43E-31

a) 5-μm AMAD particles

Based on the Figure 1 urine reference levels, a routine monitoring program for type M or S particulate 14C requires substantially greater analytical sensitivity than a monitoring program for type F, or Vapor Class SR-2 14C. For comparable intake detection of type F, M, and S particulates, monitoring by urinalysis requires analytical capabilities nominally 2, 100, and 3000 times more sensitive, respectively, than analytical methods supporting SR-2 forms. A similar comparison of fecal excretion reference levels shows that fecal sampling after the first few days is relatively insensitive to discriminating between intakes of absorption types M and S, with essentially no capability for discriminating between SR-2 and type F. Examining the ratio of same-day urine and fecal excretion would be helpful in determining particulate absorption type. Given the uncertainties in absorption type for graphite particulates associated with decommissioning activities, a prudent response to intake monitoring following workplace indications of significant intake would be to obtain both fecal and urine samples. The relatively low levels of 14C in feces from natural background environmental exposure and excretion should not pose any interference with monitoring. The same cannot be said if urine sampling is relied upon for type S monitoring, because the 0.04 dpm/mL nominal background excretion exceeds the 100-mrem reference level for urine excretion for all times post intake, and the 5-rem reference level for times greater than 180 days post intake.

While the above suggests bioassay for 14C may be quite challenging, the need for bioassay must be put in perspective with the magnitude of potential intake. Intake totaling the 350-μCi ALI of type S 14C in graphite at the 2.4 μCi/g mean concentration observed in Hanford reactor graphite, would require inhalation of 146 g of graphite. There is no realistic scenario which could result in such an intake, or for that matter a likely intake of even 1% of that magnitude, which would correspond to a dose of 50 mrem. Thus, bioassay monitoring for 14C from exposure to graphite may arguably not be a requirement. Unless source term characterization efforts show much higher concentrations than those historically observed, it would appear that the Hanford reactor decommissioning work would not require 14C bioassay.

6.0 References

10 CFR 835 Appendix A. 2007. U.S. Department of Energy, Occupational Radiation Protection. U.S. Code of Federal Regulations. Accessed August 20, 2010 at http://www.gpoaccess.gov/cfr/

Electrical Power Research Institute (EPRI). 2006. Graphite Decommissioning Options for Graphite Treatment, Recycling, or Disposal, including a discussion of Safety-Related Issues. Concord: Electrical Power Research Institute.

International Commission on Radiological Protection (ICRP). 1974. Report of the task group on Reference Man. ICRP publication 23, Pergamom Press, New York.

International Commission on Radiological Protection (ICRP). 1994. "Dose coefficients for intakes of radionuclides by workers." (ICRP publication 68.) Annals of the ICRP, 24:4, Pergamon Press, New York.

International Commission on Radiological Protection (ICRP). 1995. "Age-dependent doses to members of the public from intakes of radionuclides: Part 4 inhalation dose coefficients." (ICRP publication 71.) Annals of the ICRP, 25:3-4, Pergamon Press, New York.

International Commission on Radiological Protection (ICRP). 2001. "ICRP Database of Dose Coefficients: Workers and Members of the Public." ICRP CD-1. Elsevier Science Ltd., Pergamon Press, New York.

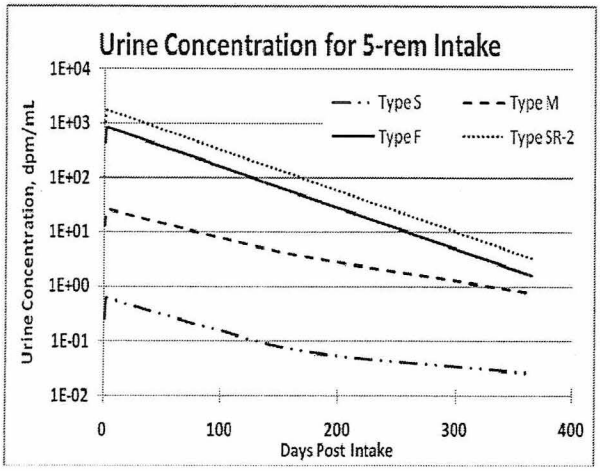
International Commission on Radiological Protection (ICRP). 2002. "Basic Anatomical and Physiological Data for Use in Radiological Protection: Reference Values." (ICRP publication 89.) Annals of the ICRP, 32:3-4, Pergamon Press, New York.

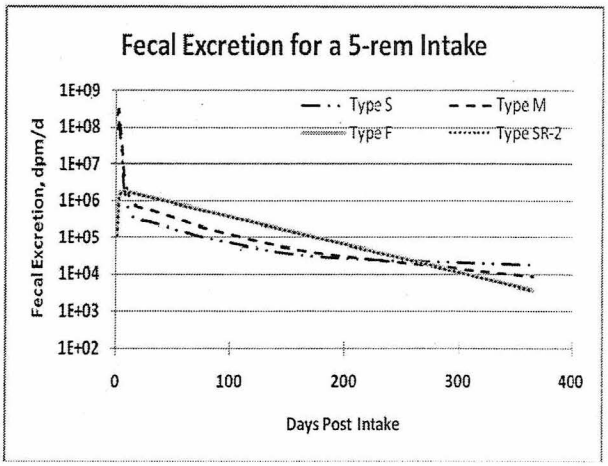
Miller R, Steffes J. Radionuclide Inventory and Source Terms for the Surplus Production Reactors at Hanford. Richland, WA: United Nuclear Corporation; UNI-3714, Rev. 1; 1987.

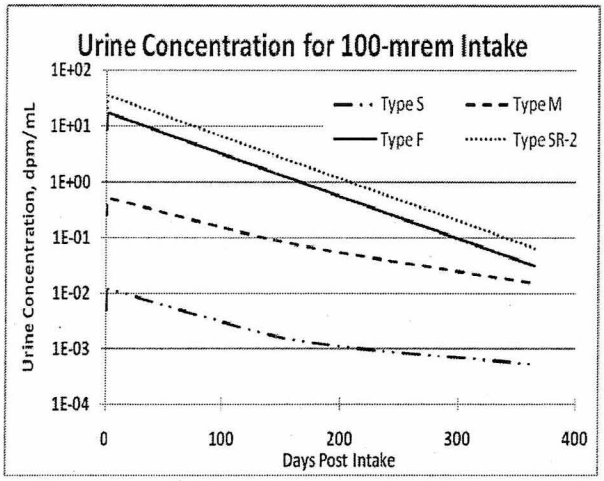
National Council on Radiation Protection and Measurements (NCRP). 1985. Carbon-14 in the Environment. NCRP Report No. 81. Bethesda, MD.

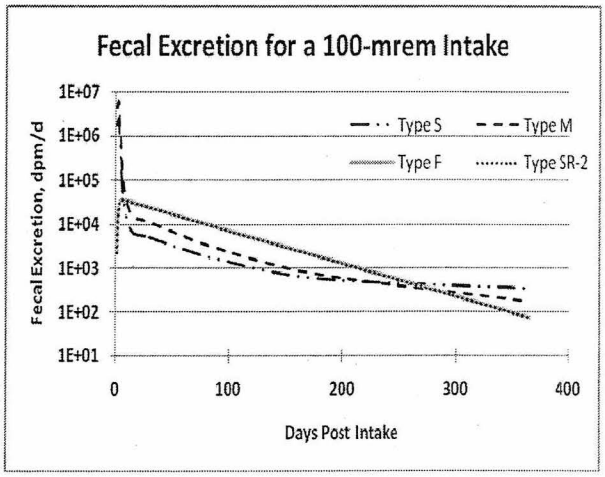
Paasch RA. 1985. A Compilation of Carbon-14 Data. Richland, WA: United Nuclear Corporation; UNI-3499.

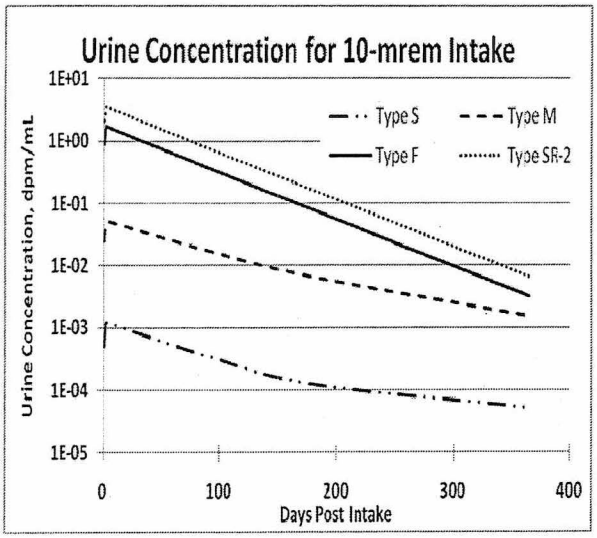
Figure 1. Urine and fecal excretion following single acute inhalation intake of ^{14}C

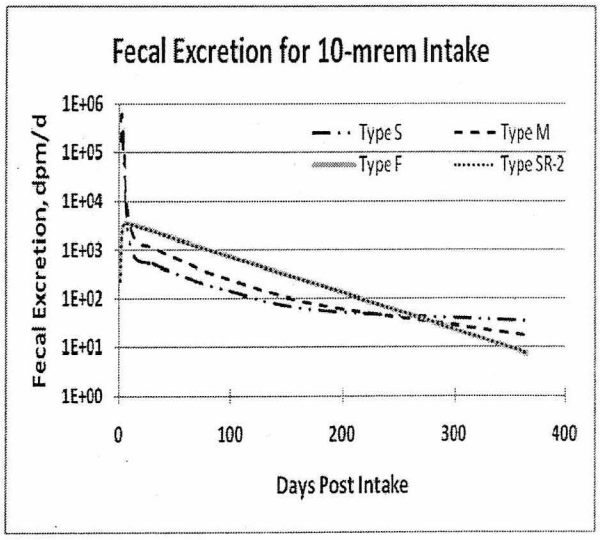












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January 17, 2011

Eugene H. Carbaugh
Pacific Northwest National Laboratory
P.O. Box 999
Richland, WA. 99352

Dear Mr. Carbaugh:

Carbon-14 Performance Evaluation Samples submitted October 26, 2010

To evaluate GEL's capabilities for C-14 urinalyses as well as follow-up GEL's failure to pass the Department of Energy's Laboratory Accreditation Program (DOELAP) category I performance testing for C-14 urinalyses, the Internal Dosimetry Program submitted 12 single blind urine samples for analysis. The samples were in 100 ml nalgene containers, 7 were spiked at 167.0 dpm/ml and 5 were blanks (Attachment 1).

On November 9, 2010 GEL reported the results (Attachment 2), activity was not detected in the five blank samples but C-14 was detected in the seven spiked samples. The MDA was estimated at 0.80 dpm/ml based on the results of the five blank samples. The seven samples that were spiked with 167.0 dpm/ml were reported with activity ranging from 131.0 through 161.0 dpm/ml. The mean relative bias was -0.091 and the mean relative precision was reported at 0.063. The Internal Dosimetry Program's (IDP) acceptance criteria for the mean relative bias is -0.20 to +0.20 and for the relative precision an absolute value less than or equal to 0.4. The contractual detection level for C-14 is 10 dpm/ml. Therefore, all the performance criteria were met and the C-14 urinalysis was determined to be acceptable.

The DOELAP performance test acceptance criteria (DOE-STD-1112-98) for the relative bias statistic is -0.25 to +0.50 and the relative precision statistic less than or equal to 0.4. A comparison of the GEL reported results with the DOELAP standards indicates that GEL likewise met the performance criteria for C-14 analyses.

The sample matrix differed from the IDP and DOELAP samples in that the IDP samples were raw urine samples and the DOELAP samples were synthetic samples using urine salts. The levels of C-14 activity in the IDP and DOELAP samples were similar. GEL technicians also observed a flaky precipitate in the DOELAP samples that they were

unable to dissolve. The precipitate in the DOELAP samples most likely contributed to GEL's relative bias ranging from -0.51 to -0.65 for the 5 DOELAP samples.

Based on GEL's performance with the twelve samples submitted by IDP, the C-14 analysis was deemed acceptable and there is high expectation that GEL will meet the DOELAP performance criteria in the 2011 retesting.

Included is the summary report from GEL, with additional emails to clarify the count times, detector efficiencies and their internal quality control results (Attachment 3). Attachment 4 is communications concerning the DOELAP samples, their preparation and the observations of GEL staff.

Sincerely,

Cheryl Antonio, CHP
Senior Research Scientist
Internal Dosimetry
Radiation & Health Technology

CLA/CLA/rab

Attachments
1. Bioassay Test Samples (8 pages)
2. QC Summary Report (1 page)
3. GEL Certificate of Analysis (29 pages)
4. Category 1 Sample Volumes (4 pages)

cc: File
LB w/o attachment

ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD_1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
C 14	1009	06	10/27/2010	10I0911	AU001	C 14	100	0.00	0.00	U		0.262	0.256	-	
C 14	1009	07	10/27/2010	10I0910	AU001	C 14	100	0.00	0.00	U		0.225	0.253	-	
C 14	1009	04	10/27/2010	10I0913	AU001	C 14	100	0.00	0.00	U		-0.007	0.263	-	
C 14	1009	11	11/06/2010	10I0918	AU001	C 14	100	0.00	0.00	U		0.232	0.246	-	
C 14	1009	03	11/06/2010	10I0914	AU001	C 14	100	0.00	0.00	U		0.386	0.249	-	
				5	U	C 14 Count		0.0000	Average Result			0.2195	DL	0.3037	Chem Yield 1.00 Time 45
								5	St Dev			0.1425	MDA	0.7998	Det Eff 0.53
C 14	1009	10	10/27/2010	10I0908	AU001	C 14	100	167.00	15.00	U		151.000	2.120	+	-0.0958
C 14	1009	08	10/27/2010	10I0912	AU001	C 14	100	167.00	15.00	U		131.000	1.850	+	-0.2156
C 14	1009	09	10/27/2010	10I0909	AU001	C 14	100	167.00	15.00	U		149.000	2.090	+	-0.1078
C 14	1009	02	11/06/2010	10I0916	AU001	C 14	100	167.00	15.00	U		160.000	2.310	+	-0.0419
C 14	1009	05	11/06/2010	10I0915	AU001	C 14	100	167.00	15.00	U		151.000	2.150	+	-0.0958
C 14	1009	01	11/06/2010	10I0917	AU001	C 14	100	167.00	15.00	U		161.000	2.300	+	-0.0359
C 14	1009	12	11/06/2010	10I0919	AU001	C 14	100	167.00	15.00	U		160.000	2.270	+	-0.0419
				7	U	C 14 Count		167.0000	Average Result			########			Mean Rel. Bias -0.0907
								7	St Dev			10.4949			Mean Rel. Precision 0.0628

Number of total U C 14 12

Total Samples 12

Total Results 12

Parameters for MDA calculation:

Detector efficiency ranged from 48% - 57%, used an average of 52.5%
Tracer Yield = 1.0, The C-14 analysis does not use a tracer
Count Time: 45 min

Attachment 2, Page 1 of 1

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Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Wednesday, February 02, 2011 10:35 AM
To: Baker, Steven C (PNNL); Barton, Clark B; Carbaugh, Eugene H; Carlson, Eric W; English, Robert G; Glines, Wayne; Haan, Thomas P; Hill, Robin L; Hilliard, James R; Jones, Robert A; Kaiser, Krista I; Kurtz, Jerry E; Lynch, Timothy P (PNNL); Rathbone, Bruce A; Ruiz, Theresa C
Cc: MacLellan, Jay; Antonio, Cheryl L; Baker, Steven C (PNNL)
Subject: GEL performance on PNNL C-14 audit samples

Attached is the summary letter and summary data report for the carbon-14 QA audit samples we submitted to GEL in October and November. GEL met our contractual performance requirements and those of HPS N13.30 (i.e., the DOELAP criteria).

Carbon 14
rfEvalSmpls Oct20

In a closely related vein, it looks like the problem that caused GEL to fail DOELAP performance testing for C-14 last year has been identified. Preliminary results of the retesting seemed to again show erratic performance by GEL that would have resulted in failure of the C-14 test. Discussions between GEL and Dave Sill at RESL (DOELAP) over the past couple of weeks have identified what appears to be a significant cause. RESL spikes with C-14 benzoate in synthetic urine and refrigerates its samples from time of preparation to time of analysis. GEL does not refrigerate their samples. In the time between receipt at GEL and analysis, microbial action within the artificial urine matrix has been found to result in degradation of the C-14 benzoate causing inaccurate results. DOELAP is now advising that samples should be refrigerated upon receipt until analysis, and GEL is instituting such a procedure. GEL will be receiving another set of test samples from DOELAP now that the problem appears to be resolved.

Samples analyzed by GEL under the PROCRAD intercomparison program had not shown any similar problem. It turns out that PROCORAD apparently acidifies their samples before sending them. That prevents the microbial action.

Our samples were not spiked using benzoate, but rather a glucose matrix. Also, we used real urine and not the synthetic urine recipe that DOELAP uses. These may be contributing factors to our samples showing better performance than the DOELAP samples.

Gene Carbaugh, CHP
Staff Scientist and Internal Dosimetry Manager
Radiation & Health Technology
Pacific Northwest National Laboratory
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P.O. Box 999, MSIN B1-60
Richland, WA 99352 USA
Tel: 509-376-6632
Fax: 509-376-8161
gene.carbaugh@pnl.gov
www.pnl.gov

Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Wednesday, February 02, 2011 10:12 AM
To: Bob Timm
Cc: MacLellan, Jay; Antonio, Cheryl L
Subject: C-14 audit data

Bob,

Attached is the summary letter and summary data page from our carbon-14 audit submittals of last October and November. From our perspective it looks like GEL has a negative bias but meets the contractual and HPS N13.30 performance criteria. I didn't provide all the other attachments, but if you want them I'll give them to you (most of the attachments was simply GEL's data report to us).

Carbon 14
erfEvalSmpls Oct20

Gene Carbaugh, CHP
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Antonio, Cheryl L

From: Carbaugh, Eugene H
Sent: Tuesday, February 01, 2011 2:18 PM
To: Bob Timm
Cc: MacLellan, Jay; Antonio, Cheryl L
Subject: RE: FW: Important Message from DOELAP

My congrats to you and Dave for the detective work. I'll buy you a beer next week!

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

From: Bob Timm [mailto:rdt@gel.com]
Sent: Tuesday, February 01, 2011 2:15 PM
To: Carbaugh, Eugene H
Cc: MacLellan, Jay; Antonio, Cheryl L
Subject: Re: FW: Important Message from DOELAP

I would be very surprised if Procorad is preserved but I haven't checked. I'll see if we still have this years lying around and test the pH.

Acidification with HNO3 would theoretically produce CO2 and losses would occur however, when I was trying to figure out our issue last year I did preserve some samples with HCL and HNO3. The HCL preserved samples didn't analyze well and I couldn't get results. The HNO3 preserved ones I got the same result as I did with unpreserved but it was still the wrong result. I didn't notice increased losses.

We analyze environmental samples unpreserved and our standards are typically in a NaOH preserved media. Sounds like refrigeration and cold shipping may be the best preservative.

Bob

On 2/1/2011 5:08 PM, Carbaugh, Eugene H wrote:
I was wondering what the impact of acidification would be on the sample, too. Does Procorad send acid-preserved samples?

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

From: Bob Timm [mailto:rdt@gel.com]
Sent: Tuesday, February 01, 2011 2:03 PM
To: Carbaugh, Eugene H
Cc: MacLellan, Jay; Antonio, Cheryl L
Subject: Re: FW: Important Message from DOELAP

Definitely.

I've been working with Dave Sill over the past week on this. I analyzed this years samples and got results all over the place like last year. I didn't want to blindly report and fail again so I called Dave. He was very open to the problem and helpful in collaborating to solve this.

We'll be adding a refrigeration requirement to our C-14 samples.

It's interesting that the Procorad samples work without refrigeration as did the samples you sent us however, the type of C-14 standard used is probably the main contributing factor.

Bob

On 2/1/2011 4:59 PM, Carbaugh, Eugene H wrote:
The below may be a contributing factor in our C-14 problem. Bob?

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

From: sillds@id.doe.gov [mailto:sillds@id.doe.gov]
Sent: Tuesday, February 01, 2011 1:47 PM
To: hickman3@llnl.gov; Carbaugh, Eugene H; raogr@ornl.gov; capotte@sandia.gov
Subject: Important Message from DOELAP

Gentlemen of the OSB,
This was some interesting detective work and I though you guys should know. Below is the e-mail that was sent to the participants.
Left me know if you have any questions....

Dear Participants,

It has come to our attention that certain laboratories are not refrigerating the DOELAP SU samples after being received in their laboratory.

The SU solutions used for C-14 analyses should be refrigerated immediately upon receipt and kept refrigerated until the C-14 analyses are completed.

If the SU solutions are left unrefrigerated for long periods of time prior to the analysis of C-14, the subsequent microbial action will lead to degradation of the C-14 labeled benzoate which will cause inaccurate analytical results for C-14.

DOELAP has verified that these solutions are stable for the entire length of the test session if they are refrigerated.

If you have any questions or if you have experienced this problem and would like a replacement set of SU samples for C-14, please contact me at the number or email listed below.

Thank You,

David Sill
Senior Technical Manager -Chemistry
U.S. Department of Energy
Radiological and Environmental Sciences Laboratory

1955 Freemont Drive, MS 4149
Idaho Falls, ID 83415
sillds@id.doe.gov
208-526-8031

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Antonio, Cheryl L

From: Bob Timm [rdt@gel.com]
Sent: Monday, June 21, 2010 10:16 AM
To: MacLellan, Jay
Cc: Carbaugh, Eugene H; stan.morton@gel.com; Antonio, Cheryl L; Jannie Shaw-Busby@gel.com; 'marletgm@id.doe.gov'
Subject: Re: TRIM: Re: FW: HANFORD INDIRECT RADIOBIOASSAY RESULTS FOR DOELAP TEST SESSION 13

PROCORAD is real urine and doesn't have the precipitate in them.

There was only one other lab in the DOELAP report. They achieved a passing result and also had a negative bias at -17%.

We still have the original samples and we are able to request new samples from DOELAP once we've figured out our issue.

H3 was performed from the same sample container and had no problems (not surprising).

Bob

On 6/21/2010 1:08 PM, MacLellan, Jay wrote:
If I remember right, the PROCORAD samples use purified urine. There wouldn't be any precipitates. It will be interesting to hear if other DOELAP participants had similar problems.

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

From: Bob Timm [mailto:rdt@gel.com]
Sent: Monday, June 21, 2010 4:25 AM
To: Carbaugh, Eugene H
Cc: stan.morton@gel.com; MacLellan, Jay; Antonio, Cheryl L; Jannie Shaw-Busby@gel.com
Subject: TRIM: Re: FW: HANFORD INDIRECT RADIOBIOASSAY RESULTS FOR DOELAP TEST SESSION 13

Thanks Gene, we also got word from DOELAP.

This was the first time we'd performed C-14 for DOELAP or at least the first time the samples had C-14 activity in them. On these specific samples, we were not able to get consistent results from one bottle to the next. There was a flaky precipitant in all bottles and I suspect it was holding a good bit of the C-14. We don't use a large enough aliquot of urine to overcome the non-homogenous nature of this sample having the precipitant.

We've performed well in the past on PROCORAD C-14 PE samples and don't have problems with Matrix Spikes and Lab Control Samples. I suspect this is more a sample matrix problem than a method problem.

We've opened a corrective action internally and we'll let you know of our findings.

Bob

On 6/18/2010 4:58 PM, Carbaugh, Eugene H wrote:
Stan, Bob, Jay,

We received the results of the DOELAP indirect radiobioassay performance testing. The transmittal to us and the full report are attached. GEL is identified as Lab 2 in the Session 13 report. GEL passed all tests except C-14 in urine, for which GEL consistently showed a negative bias ranging from -0.51 to -0.65 for the 5 samples (acceptable range for bias is -0.25 to 0.5). Lab 4 was also tested for C-14 and showed a consistent negative bias for their 5 samples of about -0.16 to -0.2.

We are not currently running C-14 as any kind of routine analysis and in fact haven't run any since 1998, so I'm not particularly worried about this. However C-14 is one of the nuclides for which we could have a potential need due to lab or cleanup work. Jay and I will be discussing this when he's back next week. At this point I just wanted to provide you with the DOELAP results.

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

From: marletgm@id.doe.gov [mailto:marletgm@id.doe.gov]
Sent: Thursday, June 17, 2010 10:14 AM
To: Baker, Steven C (PNNL)
Cc: Steve.Zobel@hq.doe.gov; Glines, Wayne M; Carbaugh, Eugene H
Subject: HANFORD INDIRECT RADIOBIOASSAY RESULTS FOR DOELAP TEST SESSION 13

Steven:

Attached is the cover letter and the indirect radiobioassay results for DOELAP Test Session 13. All performance evaluation results are listed by Lab Code. Please refer to the cover letter for your Lab Code. A quick review of performance is provided in the Summary Report section, a visual comparison of performance with the other participants is presented in the Performance Graphs section, and the result for each determination is presented in the Detailed Comparison section. Bookmarks are provided to assist in navigating through the full report. Thank you for your continued support of the DOELAP program. The DOELAP Team wishes you much success. Please contact me if you have any questions.

Best regards,

Guy

Guy M. Marlette, Chemist
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APPENDIX A

QUALITY CONTROL SAMPLE RESULTS
(Historical File Only)

ISO CD	YRMO	SEQ	ANAL DATE	TAGWORD 1	PAYID	REQ ANAL	VOL	SPIKE	UNCERT	TYPE	MR	RESULT	UNCERT	DET	REL BIAS
AM241	1101	14	01/31/2011	11A0691	YH402	IPA	145	0.0000	0.0000	F	J	-0.0115	0.0975	-	
AM241	1101	12	01/31/2011	11A0687	99200	IPA	181	0.0000	0.0000	F	J	-0.0285	0.0555	-	
AM241	1101	13	01/31/2011	11A0683	99216	IPA	114	0.0000	0.0000	F	J	-0.0246	0.0612	-	
AM241	1101	11	01/31/2011	11A0695	99159	IPA	25	0.0000	0.0000	F	J	-0.0140	0.0935	-	
AM241	1101	15	02/18/2011	11A0920	99204	IPA	99	0.0000	0.0000	F	J	0.0103	0.1100	-	
				5	**F**	**AM241 Count**		**0.0000 5**	**Average Result St Dev**		**-0.0137 0.0152**	**DL MDA**	**0.0323 0.0787**	**Chem Yield 0.86 Det Eff 0.39**	**Time 960**
AM241	1101	07	01/31/2011	11A0689	99204	IPA	212	1.6300	0.0075	F	J	1.2700	0.2800	+	-0.2209
AM241	1101	08	01/31/2011	11A0685	99217	IPA	82	1.6300	0.0075	F	J	1.3400	0.2800	+	-0.1779
AM241	1101	06	01/31/2011	11A0693	99120	IPA	88	1.6300	0.0075	F	J	1.3300	0.2800	+	-0.1840
AM241	1101	10	02/18/2011	11A0919	99217	IPA	92	1.6300	0.0075	F	J	1.7200	0.3560	+	0.0552
AM241	1101	09	02/18/2011	11A0921	99120	IPA	42	1.6300	0.0075	F	J	1.4900	0.3090	+	-0.0859
				5	**F**	**AM241 Count**		**1.6300 5**	**Average Result St Dev**		**1.4300 0.1812**			**Mean Rel. Bias Mean Rel. Precision**	**-0.1227 0.1112**
	Number of total F AM241 **10**														
PU238	1101	14	01/31/2011	11A0691	YH402	IPA	145	0.0000	0.0000	F	J	-0.0034	0.0106	-	
PU238	1101	11	01/31/2011	11A0695	99159	IPA	25	0.0000	0.0000	F	J	0.0059	0.0186	-	
PU238	1101	12	01/31/2011	11A0687	99200	IPA	181	0.0000	0.0000	F	J	0.0100	0.0092	-	
PU238	1101	13	01/31/2011	11A0683	99216	IPA	114	0.0000	0.0000	F	J	0.0086	0.0080	-	
PU238	1101	15	02/18/2011	11A0920	99204	IPA	99	0.0000	0.0000	F	J	0.0053	0.0254	-	
				5	**F**	**PU238 Count**		**0.0000 5**	**Average Result St Dev**		**0.0053 0.0052**	**DL MDA**	**0.0111 0.0368**	**Chem Yield 0.83 Det Eff 0.39**	**Time 960**
PU238	1101	08	01/31/2011	11A0685	99217	IPA	82	0.8440	0.0050	F	J	0.7160	0.0934	+	-0.1517
PU238	1101	07	01/31/2011	11A0689	99204	IPA	212	0.8440	0.0050	F	J	0.6980	0.1210	+	-0.1730
PU238	1101	06	01/31/2011	11A0693	99120	IPA	88	0.8440	0.0050	F	J	0.8470	0.1130	+	0.0036
PU238	1101	10	02/18/2011	11A0919	99217	IPA	92	0.8440	0.0050	F	J	0.8300	0.1080	+	-0.0166
PU238	1101	09	02/18/2011	11A0921	99120	IPA	42	0.8440	0.0050	F	J	0.7960	0.0998	+	-0.0569
				5	**F**	**PU238 Count**		**0.8440 5**	**Average Result St Dev**		**0.7774 0.0671**			**Mean Rel. Bias Mean Rel. Precision**	**-0.0789 0.0795**

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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
	Number of total F PU238 **10**														
PU239	1101	14	01/31/2011	11A0691	YH402	IPA	145	0.0000	0.0000	F	J	0.0052	0.0283	-	
PU239	1101	12	01/31/2011	11A0687	99200	IPA	181	0.0000	0.0000	F	J	-0.0039	0.0174	-	
PU239	1101	13	01/31/2011	11A0683	99216	IPA	114	0.0000	0.0000	F	J	-0.0071	0.0148	-	
PU239	1101	11	01/31/2011	11A0695	99159	IPA	25	0.0000	0.0000	F	J	-0.0039	0.0168	-	
PU239	1101	15	02/18/2011	11A0920	99204	IPA	99	0.0000	0.0000	F	J	0.0018	0.0321	-	
				5	**F**	**PU239 Count**		**0.0000 5**	**Average Result St Dev**			**-0.0016 0.0049**	**DL MDA**	**0.0105 0.0357**	**Chem Yield 0.83 Det Eff 0.39**	**Time 960**
PU239	1101	08	01/31/2011	11A0685	99217	IPA	82	1.0300	0.0063	F	J	0.7970	0.1010	+	-0.2262
PU239	1101	06	01/31/2011	11A0693	99120	IPA	88	1.0300	0.0063	F	J	0.7370	0.1030	+	-0.2845
PU239	1101	07	01/31/2011	11A0689	99204	IPA	212	1.0300	0.0063	F	J	0.6880	0.1210	+	-0.3320
PU239	1101	09	02/18/2011	11A0921	99120	IPA	42	1.0300	0.0063	F	J	1.0600	0.1220	+	0.0291
PU239	1101	10	02/18/2011	11A0919	99217	IPA	92	1.0300	0.0063	F	J	1.0700	0.1280	+	0.0388
				5	**F**	**PU239 Count**		**1.0300 5**	**Average Result St Dev**			**0.8704 0.1818**			**Mean Rel. Bias -0.1550 Mean Rel. Precision 0.1765**
	Number of total F PU239 **10**														
AM241	1005	16	06/03/2010	10E0313	50575	IPSA	785	0.0000	0.0000	U	L	0.0006	0.0009	-	
AM241	1005	15	06/03/2010	10E0287	91382	IPSA	1471	0.0000	0.0000	U	L	0.0011	0.0007	-	
AM241	1005	17	06/14/2010	10E0355	3C135	IPSA	1164	0.0000	0.0000	U	L	0.0054	0.0025	+	
				3	**U**	**AM241 Count**		**0.0000 3**	**Average Result St Dev**			**0.0024 0.0026**	**DL MDA**	**0.0076 0.0252**	**Chem Yield 0.87 Det Eff 0.39**	**Time 2520**
AM241	1003	05	04/21/2010	10D0067	99153	IPSA	1141	0.0200	0.0004	U	L	0.0228	0.0071	+	0.1400
AM241	1005	08	06/30/2010	10F0629	31776	IPSA	1094	0.0200	0.0003	U	L	0.0216	0.0069	+	0.0800
AM241	1005	11	06/30/2010	10F0755	99156	IPSA	1100	0.0200	0.0003	U	L	0.0210	0.0068	+	0.0500
AM241	1105	08	06/30/2010	10F0629	31776	IPSA	1094	0.0200	0.0003	U	L	0.0216	0.0069	+	0.0800
AM241	1005	09	06/30/2010	10F0656	50807	IPSA	1164	0.0200	0.0003	U	L	0.0199	0.0068	+	-0.0050
AM241	1008	06	09/07/2010	10H0554	80109	IPSA	1245	0.0200	0.0003	U	L	0.0134	0.0053	+	-0.3300
AM241	1009	17	10/29/2010	10J2409	99161	IPSA	1406	0.0200	0.0004	U	L	0.0238	0.0089	+	0.1900
AM241	1009	18	11/08/2010	10J2666	50809	IPSA	1199	0.0200	0.0004	U	L	0.0197	0.0065	+	-0.0150
AM241	1011	06	11/22/2010	10K0556	59600	IPSA	1284	0.0200	0.0003	U	L	0.0230	0.0072	+	0.1500

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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	1011	07	11/22/2010	10K0557	59698	IPSA	1102	0.0200	0.0003	U	L	0.0209	0.0067	+	0.0450
AM241	1101	04	01/29/2011	11A0233	3C142	IPSA	1174	0.0200	0.0002	U	L	0.0182	0.0062	+	-0.0900
AM241	1101	02	01/29/2011	11A0232	3C134	IPSA	1225	0.0200	0.0002	U	L	0.0165	0.0064	+	-0.1750
AM241	1101	03	01/29/2011	11A0279	99158	IPSA	1414	0.0200	0.0002	U	L	0.0243	0.0076	+	0.2150
AM241	1102	12	03/04/2011	11B0414	99150	IPSA	1180	0.0200	0.0002	U	L	0.0117	0.0054	+	-0.4150
AM241	1102	13	03/04/2011	11B0415	99154	IPSA	1265	0.0200	0.0002	U	L	0.0218	0.0072	+	0.0900
				14	**U**	**AM241 Count**		**0.0200 15**	**Average Result St Dev**			**0.0200 0.0037**			**Mean Rel. Bias 0.0007 Mean Rel. Precision 0.1832**
	Number of total U AM241 **17**														
AM243	1003	01	04/27/2010	10D0270	91386	AM243	1172	0.0000	0.0000	U		-0.0016	0.0027	-	
AM243	1007	01	08/04/2010	10G0259	80098	AM243	1160	0.0000	0.0000	U		-0.0041	0.0116	-	
AM243	1008	08	08/30/2010	10H0551	3C136	AM243	1550	0.0000	0.0000	U		0.0022	0.0038	-	
AM243	1009	19	10/01/2010	10I0209	32514	AM243	1199	0.0000	0.0000	U		-0.0007	0.0119	-	
				4	**U**	**AM243 Count**		**0.0000 4**	**Average Result St Dev**			**-0.0010 0.0026**	**DL MDA**	**0.0061 0.0186**	**Chem Yield 0.87 Det Eff 0.39 Time 2520**
	Number of total U AM243 **4**														
C 14	1009	07	10/27/2010	10I0910	AU001	C 14	100	0.0000	0.0000	U		0.2250	0.2530	-	
C 14	1009	04	10/27/2010	10I0913	AU001	C 14	100	0.0000	0.0000	U		-0.0074	0.2630	-	
C 14	1009	06	10/27/2010	10I0911	AU001	C 14	100	0.0000	0.0000	U		0.2620	0.2560	-	
C 14	1009	11	11/06/2010	10I0918	AU001	C 14	100	0.0000	0.0000	U		0.2320	0.2460	-	
C 14	1009	03	11/06/2010	10I0914	AU001	C 14	100	0.0000	0.0000	U		0.3860	0.2490	-	
				5	**U**	**C 14 Count**		**0.0000 5**	**Average Result St Dev**			**0.2195 0.1425**	**DL MDA**	**0.3037 0.7998**	**Chem Yield 1.00 Det Eff 0.53 Time 45**
C 14	1009	08	10/27/2010	10I0912	AU001	C 14	100	167.0000	15.0000	U		131.0000	1.8500	+	-0.2156
C 14	1009	10	10/27/2010	10I0908	AU001	C 14	100	167.0000	15.0000	U		151.0000	2.1200	+	-0.0958
C 14	1009	09	10/27/2010	10I0909	AU001	C 14	100	167.0000	15.0000	U		149.0000	2.0900	+	-0.1078
C 14	1009	05	11/06/2010	10I0915	AU001	C 14	100	167.0000	15.0000	U		151.0000	2.1500	+	-0.0958
C 14	1009	02	11/06/2010	10I0916	AU001	C 14	100	167.0000	15.0000	U		160.0000	2.3100	+	-0.0419
C 14	1009	01	11/06/2010	10I0917	AU001	C 14	100	167.0000	15.0000	U		161.0000	2.3000	+	-0.0359
C 14	1009	12	11/06/2010	10I0919	AU001	C 14	100	167.0000	15.0000	U		160.0000	2.2700	+	-0.0419

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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
				7	U	C 14 Count		167.0000 7	Average Result St Dev		######## 10.4949			Mean Rel. Bias Mean Rel. Precision	-0.0907 0.0628
	Number of total U C 14 12														
PU238	1003	05	04/21/2010	10D0067	99153	IPSA	1141	0.0000	0.0000	U	L	-0.0004	0.0022	-	
PU238	1005	15	06/03/2010	10E0287	91382	IPSA	1471	0.0000	0.0000	U	L	-0.0012	0.0020	-	
PU238	1005	16	06/03/2010	10E0313	50575	IPSA	785	0.0000	0.0000	U	L	0.0000	0.0017	-	
PU238	1005	17	06/14/2010	10E0355	3C135	IPSA	1164	0.0000	0.0000	U	L	-0.0007	0.0022	-	
PU238	1005	11	06/30/2010	10F0755	99156	IPSA	1100	0.0000	0.0000	U	L	0.0028	0.0037	-	
PU238	1005	08	06/30/2010	10F0629	31776	IPSA	1094	0.0000	0.0000	U	L	0.0012	0.0039	-	
PU238	1005	09	06/30/2010	10F0656	50807	IPSA	1164	0.0000	0.0000	U	L	-0.0002	0.0023	-	
PU238	1008	06	09/07/2010	10H0554	80109	IPSA	1245	0.0000	0.0000	U	L	0.0048	0.0028	-	
PU238	1009	17	10/29/2010	10J2409	99161	IPSA	1406	0.0000	0.0000	U	L	0.0000	0.0009	-	
PU238	1009	18	11/08/2010	10J2666	50809	IPSA	1199	0.0000	0.0000	U	L	0.0012	0.0011	-	
PU238	1011	06	11/22/2010	10K0556	59600	IPSA	1284	0.0000	0.0000	U	L	0.0011	0.0015	-	
PU238	1011	07	11/22/2010	10K0557	59698	IPSA	1102	0.0000	0.0000	U	L	0.0011	0.0014	-	
PU238	1101	04	01/28/2011	11A0233	3C142	IPSA	1174	0.0000	0.0000	U	L	0.0005	0.0039	-	
PU238	1101	03	01/28/2011	11A0279	99158	IPSA	1414	0.0000	0.0000	U	L	-0.0019	0.0048	-	
PU238	1101	02	01/28/2011	11A0232	3C134	IPSA	1225	0.0000	0.0000	U	L	-0.0019	0.0039	-	
PU238	1102	12	03/04/2011	11B0414	99150	IPSA	1180	0.0000	0.0000	U	L	-0.0006	0.0024	-	
PU238	1102	13	03/04/2011	11B0415	99154	IPSA	1265	0.0000	0.0000	U	L	0.0037	0.0032	-	
				17	U	PU238 Count		0.0000 17	Average Result St Dev		0.0006 0.0018	DL MDA	0.0032 0.0106	Chem Yield 0.74 Det Eff 0.39	Time 2520
	Number of total U PU238 17														
PU239	1005	15	06/03/2010	10E0287	91382	IPSA	1471	0.0000	0.0000	U	L	0.0026	0.0019	-	
PU239	1005	16	06/03/2010	10E0313	50575	IPSA	785	0.0000	0.0000	U	L	0.0003	0.0027	-	
PU239	1005	17	06/14/2010	10E0355	3C135	IPSA	1164	0.0000	0.0000	U	L	-0.0013	0.0031	-	
				3	U	PU239 Count		0.0000 3	Average Result St Dev		0.0005 0.0020	DL MDA	0.0057 0.0232	Chem Yield 0.74 Det Eff 0.39	Time 2520
PU239	1003	05	04/21/2010	10D0067	99153	IPSA	1141	0.0200	0.0005	U	L	0.0258	0.0070	+	0.2900
PU239	1005	08	06/30/2010	10F0629	31776	IPSA	1094	0.0200	0.0004	U	L	0.0101	0.0049	+	-0.4950

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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	1005	11	06/30/2010	10F0755	99156	IPSA	1100	0.0200	0.0004	U	L	0.0157	0.0067	+	-0.2150
PU239	1005	09	06/30/2010	10F0656	50807	IPSA	1164	0.0200	0.0004	U	L	0.0160	0.0058	+	-0.2000
PU239	1008	06	09/07/2010	10H0554	80109	IPSA	1245	0.0200	0.0004	U	L	0.0139	0.0047	+	-0.3050
PU239	1009	17	10/29/2010	10J2409	99161	IPSA	1406	0.0200	0.0005	U	L	0.0244	0.0064	+	0.2200
PU239	1009	18	11/08/2010	10J2666	50809	IPSA	1199	0.0200	0.0005	U	L	0.0307	0.0067	+	0.5350
PU239	1011	06	11/22/2010	10K0556	59600	IPSA	1284	0.0200	0.0004	U	L	0.0145	0.0046	+	-0.2750
PU239	1011	07	11/22/2010	10K0557	59698	IPSA	1102	0.0200	0.0004	U	L	0.0221	0.0058	+	0.1050
PU239	1101	04	01/28/2011	11A0233	3C142	IPSA	1174	0.0200	0.0003	U	L	0.0259	0.0061	+	0.2950
PU239	1101	02	01/28/2011	11A0232	3C134	IPSA	1225	0.0200	0.0003	U	L	0.0235	0.0063	+	0.1750
PU239	1101	03	01/28/2011	11A0279	99158	IPSA	1414	0.0200	0.0003	U	L	0.0223	0.0059	+	0.1150
PU239	1102	12	03/04/2011	11B0414	99150	IPSA	1180	0.0200	0.0003	U	L	0.0254	0.0059	+	0.2700
PU239	1102	13	03/04/2011	11B0415	99154	IPSA	1265	0.0200	0.0003	U	L	0.0227	0.0065	+	0.1350
				14	**U**	**PU239 Count**		**0.0200**	**Average Result**			**0.0209**			**Mean Rel. Bias 0.0464**
								14	**St Dev**			**0.0059**			**Mean Rel. Precision 0.2937**

Number of total U PU239 **17**

SR	1005	15	06/03/2010	10E0287	91382	IPSA	1471	0.0000	0.0000	U	L	0.8920	0.4610	-	
SR	1005	16	06/04/2010	10E0313	50575	IPSA	785	0.0000	0.0000	U	L	0.3310	0.4410	-	
SR	1005	17	06/11/2010	10E0355	3C135	IPSA	1164	0.0000	0.0000	U	L	1.5000	0.4430	+	
				3	**U**	**SR Count**		**0.0000**	**Average Result**			**0.9077**	**DL**	**1.7072**	**Chem Yield 0.71 Time 45**
								3	**St Dev**			**0.5847**	**MDA**	**4.1185**	**Det Eff 0.38**
SR	1003	05	04/20/2010	10D0067	99153	IPSA	1141	10.0000	0.1790	U	L	8.3400	1.1900	+	-0.1660
SR	1005	08	07/12/2010	10F0629	31776	IPSA	1094	10.0000	0.1860	U	L	8.5700	1.0600	+	-0.1430
SR	1005	11	07/12/2010	10F0755	99156	IPSA	1100	10.0000	0.1860	U	L	8.4500	1.0600	+	-0.1550
SR	1005	09	07/12/2010	10F0656	50807	IPSA	1164	10.0000	0.1860	U	L	8.0300	1.0400	+	-0.1970
SR	1008	06	09/03/2010	10H0554	80109	IPSA	1245	10.0000	0.1550	U	L	9.6100	1.2700	+	-0.0390
SR	1009	17	10/30/2010	10J2409	99161	IPSA	1406	10.0000	0.1850	U	L	10.4000	1.3500	+	0.0400
SR	1009	18	11/04/2010	10J2666	50809	IPSA	1199	10.0000	0.1850	U	L	10.7000	1.4100	+	0.0700
SR	1011	06	11/24/2010	10K0556	59600	IPSA	1284	10.0000	0.2220	U	L	9.6200	1.2200	+	-0.0380
SR	1011	07	11/24/2010	10K0557	59698	IPSA	1102	10:0000	0.2220	U	L	10.0000	1.2400	+	
SR	1101	04	02/02/2011	11A0233	3C142	IPSA	1174	10.0000	0.1200	U	L	9.6900	1.2000	+	-0.0310

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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
SR	1101	03	02/02/2011	11A0279	99158	IPSA	1414	10.0000	0.1200	U	L	10.3000	1.2600	+	0.0300
SR	1101	02	02/02/2011	11A0232	3C134	IPSA	1225	10.0000	0.1200	U	L	8.8900	1.1200	+	-0.1110
SR	1102	13	03/03/2011	11B0415	99154	IPSA	1265	10.0000	0.1510	U	L	8.9000	1.2100	+	-0.1100
SR	1102	12	03/03/2011	11B0414	99150	IPSA	1180	10.0000	0.1510	U	L	10.5000	1.4000	+	0.0500
SR	1102	14	03/29/2011	11C0284	59001	SR	1363	10.0000	0.1510	U	L	10.4000	1.2900	+	0.0400
SR	1103	09	03/30/2011	11C0244	32533	SR	1278	10.0000	0.0560	U	L	9.1100	1.3000	+	-0.0890
				16	**U**	**SR Count**		**10.0000**	**Average Result**			**9.4694**			**Mean Rel. Bias** **-0.0531**
								16	**St Dev**			**0.8701**			**Mean Rel. Precision** **0.0870**

Number of total U SR **19**

U 235	1102	08	02/24/2011	11B0183	AU001	IU	979	0.0000	0.0000	U	U	0.0010	0.0045	-
U 235	1102	09	02/24/2011	11B0186	AU003	IU	978	0.0000	0.0000	U	U	-0.0019	0.0054	-
U 235	1102	11	02/24/2011	11B0185	AU002	IU	980	0.0000	0.0000	U	U	0.0027	0.0031	-
U 235	1102	10	02/24/2011	11B0269	AU004	IU	977	0.0000	0.0000	U	U	0.0012	0.0040	-
U 235	1103	06	03/28/2011	11C0549	AU006	IU	980	0.0000	0.0000	U	U	0.0053	0.0035	-
U 235	1103	07	03/28/2011	11C0550	AU007	IU	980	0.0000	0.0000	U	U	0.0025	0.0028	-
U 235	1103	04	03/28/2011	11C0547	AU004	IU	977	0.0000	0.0000	U	U	0.0027	0.0031	-
U 235	1103	03	03/28/2011	11C0546	AU003	IU	977	0.0000	0.0000	U	U	0.0023	0.0032	-
U 235	1103	02	03/28/2011	11C0545	AU002	IU	1228	0.0000	0.0000	U	U	0.0024	0.0028	-
U 235	1011	05	03/28/2011	11C0543	AU001	IU	1228	0.0000	0.0000	U	U	0.0036	0.0047	-
U 235	1103	08	03/28/2011	11C0551	AU008	IU	981	0.0000	0.0000	U	U	0.0038	0.0037	-
U 235	1103	05	03/28/2011	11C0548	AU005	IU	977	0.0000	0.0000	U	U	-0.0003	0.0040	-
				12	**U**	**U 235 Count**		**0.0000**	**Average Result**		**0.0021**	**DL**	**0.0034**	**Chem Yield 0.87** **Time 2520**
								12	**St Dev**		**0.0019**	**MDA**	**0.0107**	**Det Eff 0.39**

Number of total U U 235 **12**

U 238	1005	14	05/14/2010	10E0380	99152	U 238	970	0.0000	0.0000	U		0.0053	0.0010	+
U 238	1005	12	05/14/2010	10E0356	3C136	U 238	978	0.0000	0.0000	U		0.0064	0.0022	+
				2	**U**	**U 238 Count**		**0.0000**	**Average Result**		**0.0058**	**DL**	**0.0050**	**Chem Yield 1.00** **Time 2520**
								2	**St Dev**		**0.0008**	**MDA**	**0.0510**	**Det Eff 0.39**
U 238	1102	10	02/24/2011	11B0269	AU004	IU	977	0.1460	0.0006	U	U	0.1670	0.0196	+ 0.1438
U 238	1102	08	02/24/2011	11B0183	AU001	IU	979	0.1460	0.0006	U	U	0.1440	0.0167	+ -0.0137

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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
U 238	1102	11	02/24/2011	11B0185	AU002	IU	980	0.1460	0.0006	U	U	0.1530	0.0182	+	0.0479
U 238	1102	09	02/24/2011	11B0186	AU003	IU	978	0.1460	0.0006	U	U	0.1360	0.0167	+	-0.0685
U 238	1103	06	03/28/2011	11C0549	AU006	IU	980	0.1460	0.0007	U	U	0.1680	0.0193	+	0.1507
U 238	1103	02	03/28/2011	11C0545	AU002	IU	1228	0.1460	0.0007	U	U	0.1570	0.0180	+	0.0753
U 238	1103	07	03/28/2011	11C0550	AU007	IU	980	0.1460	0.0007	U	U	0.1450	0.0172	+	-0.0068
U 238	1103	05	03/28/2011	11C0548	AU005	IU	977	0.1460	0.0007	U	U	0.1500	0.0174	+	0.0274
U 238	1103	08	03/28/2011	11C0551	AU008	IU	981	0.1460	0.0007	U	U	0.1470	0.0173	+	0.0068
U 238	1103	03	03/28/2011	11C0546	AU003	IU	977	0.1460	0.0007	U	U	0.1340	0.0161	+	-0.0822
U 238	1011	05	03/28/2011	11C0543	AU001	IU	1228	0.1460	0.0017	U	U	0.1380	0.0164	+	-0.0548
U 238	1103	04	03/28/2011	11C0547	AU004	IU	977	0.1460	0.0007	U	U	0.1880	0.0214	+	0.2877
				12	**U**	**U 238 Count**		**0.1460 12**	**Average Result St Dev**		**0.1522 0.0157**				**Mean Rel. Bias 0.0428 Mean Rel. Precision 0.1077**
U 238	1003	03	04/16/2010	10D0070	3G522	U 238	1392	0.2000	0.0012	U		0.3400	0.0115	+	0.7000
U 238	1005	03	05/19/2010	10E0242	SG563	U 238	1157	0.2000	0.0012	U		0.1660	0.0103	+	-0.1700
U 238	1005	04	05/26/2010	10E0729	59783	U 238	1397	0.2000	0.0012	U		0.1860	0.0087	+	-0.0700
U 238	1005	06	06/14/2010	10F0710	80109	U 238	1163	0.2000	0.0012	U		0.1740	0.0080	+	-0.1300
U 238	1007	03	07/23/2010	10G0290	99158	U 238	1378	0.2000	0.0023	U		0.1500	0.0237	+	-0.2500
U 238	1005	05	07/27/2010	10G0197	32514	U 238	1229	0.2000	0.0012	U		0.1800	0.0153	+	-0.1000
U 238	1005	07	07/27/2010	10G0207	32533	U 238	1244	0.2000	0.0012	U		0.1640	0.0202	+	-0.1800
U 238	1007	04	08/12/2010	10H0547	99161	U 238	1318	0.2000	0.0023	U		0.1730	0.0101	+	-0.1350
U 238	1008	04	08/18/2010	10H0565	59600	U 238	1380	0.2000	0.0013	U		0.1040	0.0124	+	-0.4800
U 238	1008	02	08/18/2010	10H0552	3C142	U 238	1297	0.2000	0.0013	U		0.1450	0.0059	+	-0.2750
U 238	1008	05	08/19/2010	10H0495	32472	U 238	1077	0.2000	0.0013	U		0.1590	0.0111	+	-0.2050
U 238	1009	15	09/20/2010	10I0268	3C137	U 238	1274	0.2000	0.0014	U		0.2100	0.0125	+	0.0500
U 238	1008	03	09/20/2010	10I0299	99156	U 238	1216	0.2000	0.0013	U		0.1900	0.0069	+	-0.0500
U 238	1009	14	09/20/2010	10I0267	3C135	U 238	1224	0.2000	0.0014	U		0.1890	0.0111	+	-0.0550
U 238	1009	16	10/14/2010	10J2450	3G522	U 238	1096	0.2000	0.0014	U		0.3320	0.0245	+	0.6600
U 238	1010	03	10/15/2010	10J2414	80076	U 238	1301	0.2000	0.0011	U		0.1880	0.0135	+	-0.0600
U 238	1010	02	10/15/2010	10J2356	50784	U 238	1377	0.2000	0.0011	U		0.2080	0.0127	+	0.0400
U 238	1011	04	11/15/2010	10K0494	91386	U 238	1126	0.2000	0.0023	U		0.1670	0.0080	+	-0.1650

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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	1011	03	11/15/2010	10K0558	59783	U 238	1377	0.2000	0.0023	U		0.1720	0.0152	+	-0.1400
U 238	1010	04	11/17/2010	10K0547	3C136	U 238	1119	0.2000	0.0011	U		0.2090	0.0159	+	0.0450
U 238	1011	02	11/29/2010	10K0296	SG563	U 238	1176	0.2000	0.0023	U		0.2010	0.0163	+	0.0050
U 238	1102	04	02/11/2011	11B0368	99161	U 238	1091	0.2000	0.0008	U		0.1720	0.0097	+	-0.1400
U 238	1102	03	02/11/2011	11B0369	3C142	U 238	1383	0.2000	0.0008	U		0.1550	0.0165	+	-0.2250
U 238	1102	06	02/11/2011	11B0325	32472	U 238	1319	0.2000	0.0008	U		0.1960	0.0123	+	-0.0200
U 238	1102	02	02/11/2011	11B0384	59600	U 238	1204	0.2000	0.0010	U		0.1900	0.0115	+	-0.0500
U 238	1102	05	02/28/2011	11B0270	99216	U 238	1276	0.2000	0.0008	U		0.1800	0.0118	+	-0.1000
U 238	1102	07	03/16/2011	11C0326	99156	U 238	1442	0.2000	0.0008	U		0.2140	0.0134	+	0.0700
				27	**U**	**U 238 Count**		**0.2000 27**	**Average Result St Dev**		**0.1894 0.0484**			**Mean Rel. Bias Mean Rel. Precision**	**-0.0530 0.2419**

Number of total U U 238 **41**

Total Samples **86**

Total Results **170**

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APPENDIX B

GEL QUALITY CONTROL SAMPLE REPORT SUMMARY
(Historical File Only)

PNNL
QUARTERLY
QC PACKAGE

Annual 2010
April 1, 2010 – March 31, 2011

Package Quality Control Package:

All data was packaged, reviewed, and found acceptable by the following packager:

Signature:

Name: Salina Pizarro

Date: 11-JUN-2012

Title: Analyst I

Review Quality Control Package:

All data was reviewed and found acceptable by the following Reviewer:

Signature:

Name: Robert Timm

Date: 11-JUN-2012

Title: Group Leader

Table of Contents

Sr-90 – Blank Activity/ Carrier Yield
Sr-90 – Graph for Blank Activity/ Carrier Yield
Sr-90 – LCS Bias High
Sr-90 – Graph for LCS Bias High
Sr-90 – LCS Bias Low
Sr-90 – Graph for LCS Bias Low
Th-228 – Blank Activity/ Tracer Yield
Th-228 – Graph for Blank Activity/ Tracer Yield
Th-229 – Blank Activity
Th-229 – Graph for Blank Activity
Th-230 – Blank Activity
Th-230 – Graph for Blank Activity
Th-232 – Blank Activity
Th-232 – Graph for Blank Activity
Th-232 – LCS Bias High
Th-232 – graph for LCS Bias High
Th-232 – LCS Bias Low
Th-232 – Graph for LCS Bias Low
Total Uranium – Blank Activity/Duplicate/Matrix Spike
Total Uranium – Graph for Blank Activity/Duplicate/Matrix Spike
Total Uranium – LCS Bias High
Total Uranium – Graph for LCS Bias High
Total Uranium – LCS Bias Low
Total Uranium – Graph for LCS Bias Low
Tritium – Blank Activity
Tritium – Graph for Blank Activity
Tritium – LCS Bias Low
Tritium – Graph for LCS Bias Low
U-233/234 by Alpha Spec – Blank Activity
U-233/234 by Alpha Spec – Graph for Blank Activity
U-235/236 by Alpha Spec – Blank Activity
U-235/236 by Alpha Spec – Graph for Blank Activity
U-238 by Alpha Spec – Blank Activity/Tracer Yield
U-238 by Alpha Spec – Graph for Blank Activity/Tracer Yield
U-238 by Alpha Spec – LCS Bias High
U-238 by Alpha Spec – Graph for LCS Bias High
U-238 by Alpha Spec – LCS Bias Low
U-238 by Alpha Spec – Graph for LCS Bias Low
U-236 – ICP-MS – Blank Activity/ LCS Bias High
U-236 – ICP-MS – Graph for Blank Activity/ LCS Bias High
U-233 – ICP-MS – Tracer Yield
U-233 – ICP-MS – Graph for Tracer Yield
U-236 – ICP-MS – LCS Bias Low
U-236 – ICP-MS – Graph LCS Bias Low
U-238 – ICP-MS – Blank Activity/ Duplicate/Matrix Spike
U-238 – ICP-MS – Graph for Blank Activity/ Duplicate/Matrix Spike

U-238 – ICP-MS –LCS Bias High
U-238 – ICP-MS – Graph for LCS Bias High
U-238 – ICP-MS – LCS Bias Low
U-238 – ICP-MS – Graph for LCS Bias Low

Fecal Data
Am-241 – Blank Activity/Duplicate/RER/ Tracer Yield
Am-241 – Graph for Blank Activity/Duplicate/RER/ Tracer Yield
Am-241 – LCS Bias High
Am-241 – Graph for LCS Bias High
Am-241 – LCS Bias Low
Am-241 – Graph LCS Bias Low
Cm-242 – Blank Activity/Duplicate
Cm-242 – graph for Blank Activity/Duplicate
Cm-243/244 – Blank Activity/Duplicate/Yield
Cm-243/244 – graph for Blank Activity/Duplicate/Yield
Cm-243/244 –LCS bias high
Cm-243/244 – graph for LCS bias high
Cm-243/244 –LCS bias Low
Cm-243/244 – graph for LCS bias Low
Pu-238 – Blank Activity/ RER
Pu-238 – Graph for Blank Activity/ RER
Pu-239/240 – Blank Activity/Duplicate/RER/Tracer Yield
Pu-239/240 – Graph for Blank Activity/Duplicate/RER/Tracer Yield
Pu-239/240 – LCS Bias High
Pu-239/240 – Graph for LCS Bias High
Pu-239/240 – LCS Bias Low
Pu-239/240 – Graph for LCS Bias Low

Section 3: DOELAP/NRIP RESULTS

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

Statistical 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 2010

This report summarizes Quality Control Samples (QC) analyzed with bioassay samples under Contract 112512 during the Contract Year 2010, beginning April 1, 2010 and ending March 31, 2011. Included in the report are listings for the blank, duplicate and spike results. A description of the attached data is provided below. Ten thousand six hundred and ninety-six reported samples were analyzed under this contract with a run date during the annual year. The QC samples include blanks, spikes, and duplicates.

PNNL Sample/QC Summary

Annual 2010 QC Summary Table

	Matrix	Reported Samples	QC Samples	Total Samples	%QC
Americium-Curium	FECAL	100	160	260	62
Plutonium	FECAL	95	155	250	62
Americium-Curium	URINE	1969	845	2814	30
Americium-243	URINE	27	42	69	61
Neptunium	URINE	7	15	22	68
Tritium	URINE	234	148	382	39
Thorium	URINE	17	36	53	68
Plutonium	URINE	4320	1669	5989	28
Strontium 90	URINE	1519	653	2172	30
Uranium	URINE	440	267	707	38
Uranium-236 (ICPMS)	URINE	9	24	33	73
Uranium-238 (ICPMS)	URINE	1959	750	2709	28
Total		10696	4764	15460	31

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	43	High	5.43	-0.0479	0.0728
Curium-243/244	FECAL	2	High	2.55	-0.0335	0.0191
Plutonium-239/240	FECAL	42	High	5.39	0.0051	0.0645
Americium-241	URINE	281	High	0.568	-0.0812	0.0811
Americium-243	URINE	14	High	0.455	0.00921	0.0993
Curium-243/244	URINE	68	High	0.489	-0.0951	0.0191
Neptunium-237	URINE	5	High	0.395	0.0183	0.0922
Plutonium-239/240	URINE	557	High	0.428	-0.0228	0.0891
Thorium-232	URINE	12	High	2.15	-0.0055	0.0705
Total Uranium	URINE	37	High	1.00 ug/sample	0.0072	0.0564
Uranium-238	URINE	89	High	0.381	0.0334	0.1117
Uranium-236 (ICPMS)	URINE	8	High	5760 pg/sample	-0.031	0.0501
Uranium-238 (ICPMS)	URINE	113	High	0.9926 ug/sample	-0.0139	0.0669
Strontium-90	URINE	218	High	48.3	0.0257	0.0941

CA (ICPMS)

CA

Combined bias for ^{238}U by ICPMS

$$\text{Bias} = \frac{(0.0072)(37)+(-0.0139)(113)}{37+113} = -0.008695 \approx -0.0087$$

Combined Precision ^{238}U ICPMS

$$S_B = \sqrt{\frac{(112)(0.0669)^2+(36)(0.056}{149}} = 0.0643$$

*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	43	Low	0.285	0	0.0375	0.2088
Curium-243/244	FECAL	2	Low	0.115	0	-0.0363	0.1853
Plutonium-239/240	FECAL	42	Low	0.212	0	-0.0132	0.2113
Americium-241	URINE	280	Low	0.0211	3	0.0148	0.286
Americium-243	URINE	14	Low	0.0192	0	0.0866	0.568
Curium-243/244	URINE	69	Low	0.023	0	0.143	0.268
Neptunium-237	URINE	5	Low	0.0217	0	0.19	0.297
Plutonium-239/240	URINE	555	Low	0.0217	2	0.0438	0.301
Thorium-232	URINE	12	Low	0.109	1	-0.0324	0.3097
Total Uranium	URINE	37	Low	0.05 ug/sample	0	0.1861	0.1544
Uranium-238	URINE	89	Low	0.0203	2	0.0641	0.3322
Uranium-236 (ICPMS)	URINE	8	Low	564.0301 pg/sample	0	-0.109	0.3431
Uranium-238 (ICPMS)	URINE	113	Low	0.0496 ug/sample	0	0.0618	0.1816
Strontium-90	URINE	217	Low	9.8	0	0.1214	0.1824
Tritium	URINE	74	Low	5.35 pCi/mL	0	-0.0054	0.0801

CA (ICPMS)

Combined Bias and Precision for ^{238}U by ICPMS

CA

$$\text{Bias} = \frac{(0.1861)(37)+(0.0618)(113)}{37+113} = 0.0925$$

$$\text{Precision} = \sqrt{\frac{(36)(0.1544)^2+(112)(0.1816)^2}{149}} = 0.175$$

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 quarter. The Strontium MDAs are adjusted according to the average tracer yield for the quarter. The Uranium by ICP-MS MDAs are based on the standard deviation of a standard analyzed each day when samples are analyzed throughout the quarter.

PNNL
Annual 2010

Isotope	Matrix	N#	MDA	Lc	Avg. Volume	Sample units	Detector Yield	Count Efficiency	Time (min)
Am-241	Urine	281	0.018	0.00926	1	dpm/s	0.869	0.391	2520
Am-243	Urine	14	0.021	0.01139	1	dpm/s	0.867	0.391	2520
Cm-242	Urine	71	0.009	0.00375	1	dpm/s	0.869	0.391	2520
Cm-243/244	Urine	71	0.010	0.00419	1	dpm/s	0.869	0.391	2520
Np-237	Urine	5	0.031	0.01852	1	dpm/s	0.6484	0.391	2520
Pu-238	Urine	557	0.012	0.00530	1	dpm/s	0.76	0.391	2520
Pu-239/240	Urine	557	0.017	0.00809	1	dpm/s	0.76	0.391	2520
Th-228	Urine	12	0.071	0.05392	1	dpm/s	0.765	0.386	2520
Th-229	Urine	12	0.038	0.02569	1	dpm/s	0.765	0.386	2520
Th-230	Urine	12	0.051	0.03515	1	dpm/s	0.765	0.386	2520
Th-232	Urine	12	0.031	0.01958	1	dpm/s	0.765	0.386	2520
U-233/234	Urine	89	0.032	0.01772	1	dpm/s	0.801	0.386	2520
U-235/236	Urine	89	0.018	0.00866	1	dpm/s	0.801	0.386	2520
U-238	Urine	89	0.031	0.01737	1	dpm/s	0.801	0.386	2520
Sr-90	Urine	218	4.650	0.72917	1	dpm/s	0.707	0.379	45
Tritium	Urine	74	0.808	0.50502	0.01 L	dpm/L	n/a	0.243	20
U-236 (ICPMS)	Urine	8	36.408	1.2575	0.5	pg/s	0.870	n/a	n/a
U-238 (ICPMS)*^	Urine	150	0.021	0.0062	0.001 L	ug/s	n/a	n/a	n/a
Am-241	Fecal	43	0.105	0.04276	0.3333	dpm/s	0.864	0.391	960
Cm-242	Fecal	2	0.043	0.00689	0.3333	dpm/s	0.864	0.391	960
Cm-243/244	Fecal	2	0.048	0.00999	0.3333	dpm/s	0.864	0.391	960
Pu-238	Fecal	42	0.060	0.01582	0.3333	dpm/s	0.827	0.391	960
Pu-239/240	Fecal	42	0.096	0.03562	0.3333	dpm/s	0.827	0.391	960

*U-238 (ICPMS) MDA uses a 2:15 dilution factor.
^U-238 (ICPMS) contains both U-238 by ICPMS and Total Uranium data, by ICPMS ct

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.

*Unless otherwise noted.

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	RER	TPU	Parent Sample	Result	TPU
1	1202250865	1726	05-NOV-10	0.703	0.828	0.0277	265727001	0.0497 and 0.0217	0.0277 and0 .0194
2	1202251487	1707	09-NOV-10	0.821	0.477	0.0156	265781001	0.0156 and -0.00813	0.0156 and 0.0472
3	1202257351	1721	15-NOV-10	0.851	0.0495	0.0151	266510001	-0.00463 and -0.00349	0.0151 and 0.0174
4	1202292553	1687	31-DEC-10	0.823	0.249	0.0764	269159001	-0.000765 and 0.0187	0.0764 and 0.0172
5	1202295770	1630	08-JAN-11	0.961	0.11	0.0963	269414001	-0.00404 and -0.0169	0.0963 and 0.0669
6	1202301857	1644	31-JAN-11	0.795	0.65	0.0186	270160001	0.017 and -0.0246	0.0186 and 0.0612
7	1202307613	1707	22-JAN-11	0.918	0.158	0.0159	270577001	0.0137 and 0.0101	0.0159 and 0.0163
8	1202314736	1647	05-FEB-11	0.872	0.0706	0.122	271226001	-.00399 and -.0147	0.122 and 0.0903
9	1202319478	1659	10-FEB-11	0.956	0.0626	0.0952	271557001	-0.00195 and 0.00764	0.0952 and 0.12
10	1202321667	1671	14-FEB-11	0.973	0.225	0.0464	271775001	-0.0246 and 0.00525	0.0464 and 0.124
11	1202324645	1692	18-FEB-11	0.856	0.247	0.331	272001001	1.6 and 1.72	0.331 and 0.356
12	1202329005	1623	25-FEB-11	0.79	0.627	1.11	272391001	5.67 and 4.76	1.11 and 0.935
13	1202333377	1670	02-MAR-11	0.881	0.767	0.0256	272666001	0.0521 and 0.0277	0.0256 and 0.0189
14	1202336456	1625	08-MAR-11	0.729	0.147	0.0367	272893001	-0.0115 and -0.00173	0.0367 and 0.0554
15	1202345364	1623	19-MAR-11	0.694	0.877	0.0151	273714001	0.0226 and 0.0483	0.0151 and 0.0251
16	1202353302	1641	29-MAR-11	0.829	0.426	0.0284	274310001	-0.0316 and 0.00431	0.0284 and 0.0793
17	1202119811	1636	26-MAY-10	0.777	2.08	00.0114	253072001	0.0216 and -0.00295	0.0114 and 0.00298
18	1202150092	1628	07-JUL-10	0.82	0.528	0.0233	255420001	-0.00296 and 0.0109	0.0233 and 0.0121
19	1202151892	1715	07-JUL-10	0.863	0.498	0.0331	255478001	0.00158 and -0.0164	0.0331 and 0.0145
20	1202155654	1629	13-JUL-10	0.62	0.866	0.0897	255826001	0.322 and 0.227	0.0897 and 0.0631
21	1202158199	1719	16-JUL-10	0.892	0.347	0.0103	256048001	0.0119 and 0.017	0.0103 and 0.0105
22	1202162244	1714	22-JUL-10	0.857	1.56	0.0172	256414001	0.0318 and 0.00347	0.0172 and 0.00589
23	1202168048	1722	30-JUL-10	0.79	1.51	0.012	256841001	0.0148 and 0.0554	0.012 and 0.024
24	1202173014	1635	05-AUG-10	0.997	0.207	0.0109	257280001	0.0134 and 0.0104	0.0109 and 0.00954
25	1202201073	1720	01-SEP-10	0.765	0.489	0.00805	259686001	0.00396 and -0.00649	0.00805 and 0.0198
26	1202204061	1702	08-SEP-10	0.698	0.542	0.165	259834001	0.717 and 0.853	0.165 and 0.189
27	1202213425	1688	21-SEP-10	0.916	0.125	0.0546	260573001	0.193 and 0.203	0.0546 and 0.0582
28	1202215029	1714	21-SEP-10	0.827	0.549	0.0434	260690001	0.132 and 0.101	0.0434 and 0.0361
29	1202218896	1701	27-SEP-10	0.782	0.304	0.00975	261055001	0.00408 and 0.00869	0.00975 and 0.0116
30	1202247800	1643	04-NOV-10	0.774	01.5	0.0201	265418001	-0.0222 and 0.0157	0.0201 and 0.0154

31	1202247987	1672	04-NOV-10	0.816	0.235	0.0288	265423001	-0.0152 and -0.0029	0.0288 and 0.0438

Plutonium-238									
#	Sample ID	Inst	Run Date	Tracer Yield	RER	TPU	Parent Sample	Result	TPU
1	1202292563	1692	31-DEC-10	0.91	1.78	0.0026	269159001	-0.00678 and 0	0.0026 and 0.00277
2	1202295774	1623	08-JAN-11	0.959	1.1	0.0103	269414001	0.0173 and 0.00515	0.0103 and 0.00386
3	1202301861	1655	31-JAN-11	0.91	0.622	0.0121	270160001	0.0176 and 0.00857	0.0121 and 0.00801
4	1202307617	1714	22-JAN-11	0.851	1.71	0.00674	270577001	0.00891 and -0.00331	0.00674 and 0.00237
5	1202314740	1652	05-FEB-11	0.906	0.652	0.0111	271226001	0 and 0.00911	0.0111 and 0.00848
6	1202319482	1664	10-FEB-11	0.976	0.256	0.0144	271557001	0.00454 and 0	0.0144 and 0.0104
7	1202321671	1676	14-FEB-11	0.899	1.08	0.0175	271775001	-0.00177 and 0.0251	0.0175 and 0.0177
8	1202324649	1685	18-FEB-11	0.861	0.951	0.0937	272003001	0.694 and 0.796	0.0937 and 0.0998
9	1202329009	1629	25-FEB-11	1.05	0.138	0.0816	272391001	0.629 and 0.613	0.0816 and 0.0818
10	1202333387	1675	02-MAR-11	0.878	0.577	0.0253	272666001	0.00735 and -0.00947	0.0253 and 0.0145
11	1202336464	1713	08-MAR-11	00.763	0.299	0.0244	272893001	-0.00108 and -0.00954	0.0244 and 0.0144
12	1202345368	1629	19-MAR-11	00.597	0.728	0.0169	273714001	-0.0107 and 0.0108	0.0169 and 0.0242
13	1202353306	1646	29-MAR-11	0.81	0.44	0.0194	274310001	-0.0153 and -0.00399	0.0194 and 0.0169
14	1202251491	1713	09-NOV-10	0.819	0	0.00342	265781001	0 and 0	0.00342 and 0.00363
15	1202257355	1726	15-NOV-10	0.756	0.439	0.00594	266510001	-0.00115 and -0.00459	0.00594 and 0.00512
16	1202119815	1641	26-MAY-10	0.475	0.507	0.0134	253072001	0.0148 and 0.0261	0.0134 and 0.0178
17	1202150096	1680	07-JUL-10	0.735	0	0.00595	255420001	-0.000973 and -0.000973	0.00595 and 0.00542
18	1202151902	1726	07-JUL-10	0.884	0.00719	0.0049	255478001	-0.00441 and -0.00436	0.0049 and 0.00494
19	1202155661	1638	13-JUL-10	0.362	0.52	0.01	255826001	-.000942 and -.00795	0.01 and 0.00903
20	1202158203	1724	16-JUL-10	0.95	0.613	0.00404	256048001	-0.000271 and -0.00417	0.00404 and 0.00491
21	1202162248	1709	22-JUL-10	0.793	0.661	0.00407	256414001	0 and -0.00364	0.00407 and 0.00371
22	1202173026	1639	05-AUG-10	0.832	01.46	0.00784	257280001	0.00907 and -0.00322	0.00784 and 0.00311
23	1202201077	1726	01-SEP-10	0.965	0.733	0.00327	259686002	0 and 0.00451	0.00327 and 0.00521
24	1202204065	1710	08-SEP-10	0.683	0.513	0.0142	259834001	0.0178 and 0.03	0.0142 and 0.0191
25	1202213436	1692	21-SEP-10	0.921	0.424	0.00608	260573001	0.00788 and 0.0126	0.00608 and 0.00932
26	1202215033	1694	21-SEP-10	0.682	0.702	0.00473	260690001	-0.00452 and 0	0.00473 and 0.00437
27	1202218900	1695	27-SEP-10	.689	.563	.0084	261055001	0.0108 and 0.019	0.0084 and 0.0119
28	1202247815	1654	04-NOV-10	.502	0	.00592	265418001	0 and 0	0.00592 and 0.00784
29	1202250869	1679	05-NOV-10	.464	1.35	.00693	265727001	-0.0124 and 0	0.00693 and 0.00603

Plutonium-239/240									
#	Sample ID	Inst	Run Date	Tracer Yield	RER	TPU	Parent Sample	Result	TPU
1	1202251491	1713	09-NOV-10	0.819	0.703	0.014	265781001	-0.0176 and -0.000426	0.014 and 0.02
2	1202257355	1726	15-NOV-10	0.756	0.701	0.0139	266510001	0.0155 and 0.000704	0.0139 and 0.0159
3	1202292563	1692	31-DEC-10	0.91	0.582	0.0125	269159001	0.0083 and -0.00393	0.0125 and 0.0169
4	1202295774	1623	08-JAN-11	0.959	1.23	0.0149	269414001	0.0137 and -0.00879	0.0149 and 0.0107
5	1202301861	1655	31-JAN-11	0.91	0.388	0.0333	270160001	0.00702 and -0.00711	0.0333 and 0.0148
6	1202307617	1714	22-JAN-11	0.851	0.25	0.00965	270577001	-0.00419 and -0.0075	0.00965 and 0.00904
7	1202314740	1652	05-FEB-11	0.906	0.246	0.0179	271226001	0.0312 and 0.0379	0.0179 and 0.0206
8	1202319482	1664	10-FEB-11	0.976	0.0698	0.0404	271557001	0.00524 and 0.00161	0.0404 and 0.0327
9	1202321671	1676	14-FEB-11	0.899	0.765	0.0167	271775001	0.016 and -0.00611	0.0167 and 0.0236
10	1202324649	1685	18-FEB-11	0.861	1.47	0.105	272003001	0.826 and 1.06	0.105 and 0.122
11	1202329009	1629	25-FEB-11	1.05	0.6	0.794	272391001	11.5 and 12.2	0.794 and 0.855
12	1202333387	1675	02-MAR-11	0.878	1.22	0.0254	272666001	0.0537 and 0.0166	0.0254 and 0.0165
13	1202336464	1713	08-MAR-11	0.763	0.421	0.0173	272893001	.0198 and .0105	0.0173 and 0.0137
14	1202345368	1629	19-MAR-11	0.597	0.247	0.0163	273714001	-0.00545 and -0.000173	0.0163 and 0.0138
15	1202353306	1646	29-MAR-11	0.81	0.163	0.0171	274310001	0.0181 and 0.00689	0.0171 and 0.0664
16	1202119815	1641	26-MAY-10	0.475	0.623	0.0263	253072001	-0.00849 and 0.00959	0.0263 and 0.0123
17	1202150096	1680	07-JUL-10	0.735	0.232	0.0212	255420001	0.0332 and 0.027	0.0212 and 0.0163
18	1202151902	1726	07-JUL-10	0.884	0.85	0.0179	255478001	0.0239 and 0.00704	0.0179 and 0.00856
19	1202155661	1638	13-JUL-10	0.362	0.0902	.0191	255826001	0.018 and 0.0157	0.0191 and 0.0169
20	1202158203	1724	16-JUL-10	0.95	0.332	0.0119	256048001	0.00192 and -0.00303	0.0119 and 0.00896
21	1202162248	1709	22-JUL-10	0.793	1.63	0.0134	256414001	0.012 and 0.0591	0.0134 and 0.0256
22	1202173026	1639	05-AUG-10	0.832	0.135	0.0214	257280001	0.00253 and -0.00132	0.0214 and 0.0189
23	1202201077	1726	01-SEP-10	0.965	0.0722	0.0324	259686002	-0.0042 and -0.00733	0.0324 and 0.0288
24	1202204065	1710	08-SEP-10	0.683	0.627	0.14	259834001	1.14 and 1.27	0.14 and 0.153
25	1202213436	1692	21-SEP-10	0.921	0.51	0.0673	260573001	0.443 and 0.493	0.0673 and 0.0714
26	1202215033	1694	21-SEP-10	0.682	0.255	0.0355	260690001	-0.000191 and 0.00981	0.0355 and 0.0168
27	1202218900	1695	27-SEP-10	0.689	0.353	0.0216	261055001	-0.0217 and -0.00763	0.0216 and 0.0335
28	1202247815	1654	04-NOV-10	0.502	0.298	0.0444	265418001	0.000724 and -0.0148	0.0444 and 0.0274
29	1202250869	1679	05-NOV-10	0.464	1.55	0.0282	265727001	0.0362 and -0.0182	0.0282 and 0.021

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

Three Americium blanks are denoted as outliers; however, the results are less than the RDL of 0.02 dpm/sample.

Out of two thousand eight hundred and fourteen yields, thirty-eight are denoted as outliers. Ten (0.36%) are less than the low of 40%. Fourteen (0.46%) are less than the minimum of 20%.

Out of two hundred and eighty-one Am-241 high LCS's, two (0.71%) are less than 75%, and one is denoted as an outlier. One (0.36%) is greater than 125% and is denoted as an outlier. There is one more high LCS than low LCS due to the sample 1202073259 not having any recovery or yield (in DUSE).

Out of two-hundred and eighty Am-241 Low LCS's, forty-nine (17.50%) are less than 75%. Fifty-one (18.21%) are greater than 125%. One is denoted as an outlier.

Out of fourteen Am-243 Low LCS's, six (42.86%) are less than 75%. Four (28.57%) are greater than 125%.

One Curium-242 blank is denoted as an outlier; however, the result is less than the RDL of 0.02 dpm/sample.

Out of seven hundred and eighteen Curium yields, five are denoted as outliers. Two (0.28%) are less than 40%, and three (0.42%) are less than the minimum of 20%.

Out of sixty-nine low Curium LCS's, four (5.80%) are less than 75%. Twenty-four (34.8%) are greater than 125%.

There is one less high Curium LCS than low LCS due to the LCS 1202073259 not having any recovery or yield (in DUSE).

There are two more Curium blanks than LCS's due to the Curium LCS's (1202141328, 1202141329, 1202197792, and 1202197793) were not spiked for the Curium portion of the Americium/Curium batch. The Americium portion was spiked.

For the QC Summary Table, the Urine Americium-Curium numbers are derived from Americium/Curium section of the database results from the combined Americium/Curium batches plus the single batch of Curium only analysis. From the Americium/Curium combined batches: 281 MB+281 High LCS+280 Low LCS=842 QC samples. There are 2802 Am/Cm yields so 2802-842=1960 Reported samples. The Curium only batch has 1MB+1High LCS+1Low LCS=3 QC samples. There are 12 Cm only yields total. 12 total samples – 3 QC samples =9 Cm only Reported Samples. Finally, the two sets of numbers (1 from Am/Cm combined batches and 1 from the Cm only batch) were added together to get the final numbers.

Reported Samples = 9 Cm only +1960 Am/Cm combined batch=1969

QC Samples=3 Cm only + 842 Am/Cm combined batch=845

Total Samples= 12 Cm only + 2802 Am/Cm combined batch=2814

Neptunium

The MDA for Neptunium-237 is greater than the RDL of 0.02 dpm/sample; however, the Contract Limit is satisfied per the SOW because 100% of the results spiked at contractual decision limit were greater than the decision level.

Out of twenty-two Neptunium yields, one (4.55%) is less than the low of 50%.

Out of five Neptunium Low LCS's, three (60.00%) are greater than 125%.

Plutonium

Eight Plutonium-238 blanks are denoted as outliers; however, the results are less than the RDL

Six Plutonium-239/240 blanks are denoted as outliers; however, the results are less than the RDL.

Out of five thousand nine hundred and eighty-nine Plutonium yields, seventy-five (1.25%) are less than the minimum of 25%. Three hundred and thirty-nine (5.66%) were less than the low of 50%. Sixty-two are denoted as outliers.

Out of five hundred and fifty-eight Plutonium high LCS's, six (1.08%) are less than 75%. One (0.18%) is greater than 125%. Five are denoted as outliers. One Plutonium high LCS is in DUSE.

Out of five hundred and fifty-eight Plutonium Low LCS's, eighty (14.34%) are less than 75%. One hundred and twenty-five (22.40%) are greater than 125%. Two are denoted as outliers. One Plutonium low LCS is in DUSE.

There are two more high level Plutonium LCS than low level. Sample 1202072720 was lost during the prep phase along with the Strontium portion. Sample 1202218915 had a failing high tracer yield resulting from a low LCS recovery.

Strontium

Two Strontium blanks are denoted as outliers; however, the results are less than the RDL of 10 dpm/sample.

Out of two thousand one hundred and seventy-two Strontium yields, ten (0.46%) are less than the minimum of 25%. One hundred and twenty-two (5.62%) were less than the low yield of 50%. Eleven are denoted as outliers.

Out of two hundred and nineteen Strontium high LCS's, one (0.46%) is less than 75% and is denoted as an outlier. One (0.46%) is greater than 125%. One Strontium high LCS is in DUSE.

Out of two hundred and nineteen Strontium low LCS's, two (0.91%) are less than 75%. Forty-three (19.63%) are greater than 125%. Two are denoted as outliers. One Strontium low LCS is in DUSE.

There is one less low Strontium LCS than high level due to sample 1202072744 being lost (along with the Plutonium) during the prep phase.

Thorium

One Thorium-228, and Th-230 MB is greater than the RDL and is denoted as an outlier; however, this is due to a low tracer yield.

Out of fifty-three thorium yields, four (7.55%) are denoted as outliers and are less than the minimum of 20%. Two (3.77%) are less than the low yield of 50%. One is denoted as an outlier.

One Thorium-229 and one Thorium232 MB is denoted as an outlier; however, the result is less than the RDL of 0.1 dpm/sample.

Out of twelve Thorium Low LCS's, one (8.33%) is less than 75% and is denoted as an outlier. This is due to the low tracer recovery documented by DER 912592 (batch 1057630).

Tritium

There are no observations for Tritium for this year.

Uranium

One Uranium-235/236 MB is denoted as an outlier; however, the result is less than the RDL of 0.02 dpm/sample.

Out of seven hundred and seven Uranium yields, five (0.71%) are less than the low of 40%, and four are denoted as outliers. Twenty (2.83%) were less than the minimum of 20%.

Out of ninety-one high LCS's, one (1.10%) is less than 75% and is denoted as an outlier. Three (3.30%) are greater than 125%. Two Uranium high LCS's are in DUSE.

Out of ninety-one Low LCS's, fifteen (16.48%) are less than 75%. Twenty-five (27.47%) are greater than 125%. One is denoted as an outlier. Two Uranium low LCS's are in DUSE.

The MDA's for Uranium-233/234 and Uranium-238 are greater than the RDL of 0.02 dpm/sample due to elevated background levels; however, the Urnaium-235 MDA is below the RDL.

Uranium-236 by ICPMS

Out of thirty-three Uranium-233 yields, one (3.03%) is less than the minimum of 15% and is denoted as an outlier. This is due to a prep error that resulted in low tracer yield.

Uranium-238 by ICPMS and Total Uranium

Three Uranium-238 by ICPMS MBs are denoted as outliers; however, the results are less than the RDL of 0.06 ug/sample.

Two Uranium-238 by ICPMS matrix spikes are greater than the recovery requirements of 75%-125%, one is denoted as an outlier. One of the results was greater than five times the spike, and the spike recovery is not applicable. The other result is outside of GEL's requirement; however, the result was within the relative bias as set by ANSI 13:30 (-0.25 to +0.5).

Three Uranium-238 by ICPMS duplicates are denoted as outliers; however, the results of the parent sample and duplicate are less than 5 times the MDA, and the RPDs are less than the requirement of 100%.

Out of one hundred and fifty (113 U-238 by ICPMS +37 T.U.) Uranium-238 by ICPMS and Total Uranium high LCS's, one (0.66%) is greater than 125%, and four are denoted as outliers.

Out of one hundred and fifty (113 U-238 by ICPMS +37 T.U.) Uranium-238 by ICPMS and Total Uranium low LCS's, fifteen (3 from U-238 by ICPMS+12 from Total Uranium) (10.00%) are less than 75%. Thirteen (8.67%) are greater than 125%, and one is denoted as an outlier.

Note that the N# on the MDA study is 150 (113 MB from U-238 by ICPMS + 37 MB from T.U.).

Note that the QC Summary Table's Reported Samples changed. There were 1573 samples reported in the LIMS systems as Uranium-238 (by ICPMS) and 386 samples reported as Total Uranium. The total is 1959. The QC Samples also changed. There were 565 QC Samples reported as Uranium-238 (by ICPMS), and 185 QC samples reported as Total Uranium. The total QC sample is 750.

Fecal

Americium

One Americium-241 RER is denoted as an outlier; however, the result is less than 3.

One Am-241 duplicate is denoted as an outlier; however, the results of the parent sample and duplicate are less than 5 times the MDA and are less than the requirement of 100%.

Out of two hundred and sixty Americium yields, three (1.15%) are less than the low of 40% and are denoted as outliers. There are 256 yields for Am/Cm batch, 160 QC Samples, and 96 Reported samples (256-160=96). Out of the Am/Cm batch, 4 samples were Cm only samples. So the Reported Samples will be 96 Am/Cm combined reported samples + 4 Cm only = 100. The Total Samples is 256+4=260. The QC sample numbers do not change because the Cm only samples were prepped in Am/Cm combined batch, and the QC numbers have already been accounted for.

Out of forty-three Am-241 low LCS's, three (6.98%) are less than 75%. Seven (16.28%) are greater than 125%.

Curium

There are no variations for Curium-242 MB or duplicate graphs due to samples having the same results.

Plutonium

Two Plutonium-238 blanks are denoted as outliers; however, the results are less than the RDL of 0.2 dpm/sample.

One Plutonium-239/240 duplicate is greater than 20% and is denoted as an outlier; however, the RER is less than 3.

Out of two hundred and fifty Plutonium yields, fifteen (6.00%) are less than the low of 50%. Five are denoted as outliers.

Out of forty-two Plutonium low LCS's, one (2.38%) is less than 75%. Five (11.90%) are greater than 125%.

NRIP/DOELAP

GEL's results are Lab 2 for DOELAP.

The DOELAP results are sent in a separate package labeled DOELAP.

Incident Reports

Incident associated with Work Order 250144, April 19, 2010

The incident involved a client requested data recheck on the following result: Tagword 10C0877 was reported positive for Pu-239. The sample ID is 250144002.

The data was reviewed by the Group Leader, Bob Timm. Hand calculations of the results confirm. The Pu-239/240 region of interest has 66 net counts over the 42 hour counting period. The background is 1 count. A short 16 hour count was performed to confirm the activity that resulted in 30 net counts in 16 hours so the result confirms with a recount. The sample was in a three sample batch with a blank, high LCS and low LCS. The other two samples in the batch did not have activity present. The matrix blank did not have activity. The high LCS had a recovery of 83.5% and the low LCS had a recovery of 132%. All recoveries and tracer yields were as expected.

This incident is considered closed.

Incident associated with Work Order 250211, April 21, 2010

The incident involved a client requested data recheck on results for four tagwords (10C0134, 10C0294, 10C0598, 10C0897) that were reported positive for Pu-239, and 10C0146 was positive for Pu-238. They were all routine analyses and received about the same time. The GEL sample IDs are 250211001, 250211003, 250211005, 250542007, and 250211002 respectively.

GEL Laboratories (GEL) reviewed the radiobioassay sample results for 10C0134 (250211001), 10C0146 (250211002), 10C0294 (250211003), 10C0598 (250211005), and 10C0667 (250542007). After review by the Group Leader Bob Timm, the following comments were provided regarding these samples: ·Sample 10C0134 has nine net counts scattered throughout the Pu-239 region of interest (ROI) and has a background of zero counts. ·Sample 10C0294 has ten net counts scattered throughout the Pu-239 ROI and has a background of zero counts. ·Sample 10C0598 has seven net counts scattered throughout the Pu-239 ROI and has a background of one count. ·Sample 10C0146 has six net counts scattered throughout the Pu-238 ROI and has a background of zero counts. Samples 10C0134, 10C0294, 10C0598 and 10C0146 were prepared and analyzed within the same batches. If the samples required fewer counts, this would have made these results below the action level. The Method Blank and five other samples in the batch do not show positive detects. The LCS recovery is 97% and all other quality control criteria are acceptable. Compared to the other samples in the batch, the spectra for the above samples look a little noisier. There appears to be more erroneous counts outside the plutonium ROI than in the other spectra. This may be due to detector noise or something associated with the chemistry utilized with the procedure. Sample 10C0897 was in a batch containing eight samples. The Method Blank and eight other samples in the batch do not show positive detects. The LCS recovery is 89.7% and all other quality control criteria are acceptable. For this sample, there are 17 net counts with a zero background. A peak begins to form in the ROI where you would expect for Pu-239. This sample appears to contain Pu-239 activity based on the spectral data.

This incident is considered closed.

Incident associated with Work Order 254190, June 29, 2010

The incident involved tagword 10F0447 requested for Neptunium-237 analysis. The analyst Kristi Williams analyzed the batch and has been qualified since May 2010. To investigate if the tracer failure was due to the procedure or reagents, she analyzed synthetic urine samples to verify whether either of these were the source of the error. Ms. Williams prepared and analyzed four LCS to verify the method and per performance. She achieved an average 100% tracer recovery and a 92% LCS recovery. This leads GEL to the conclusion that there was an analyst error in performing the original analysis. It was also noted by the Group Leader Bob Timm that a checklist was not available to help assist the analyst with the steps through the process. In resolution, a checklist was created and validated for *The Determination of Neptunium in Urine* (GL-RAD-B-017).

This corrective action is considered closed.

Incident associated with Work Order 245645, August 04, 2010

The incident involved a discussion of the possible causes for failing the carbon-14 DOELAP performance test. Because DOELAP accreditation is a condition in the contract SOW, an incident report was requested. No issue was taken with the facts that were provided previously, but a formal report was requested.

Group Leader Robert Timm reviewed the data associated with samples DL1SU0110LB-1 through DL1SU0110LB-6 (Work Order 245945, SDG DLAP0110SUH3C14). No plausible errors could be determined from the data. A description of the samples included that an insoluble material is in the sample provided by DOELAP. GEL suspected that the material may have contained some of the carbon-14 spike. Due to the limited aliquot taken for analysis, with the unknown material the sample, a homogeneous sample was difficult to obtain and this may be the reason for the erroneous results and failures. Laboratory staff attempted to dissolve the material by preserving the samples to a pH>2 with both Nitric Acid and Hydrochloric Acid. Neither acid aided in dissolving the material.

Upon investigation, an additional test on the original DOELAP samples was performed. Three samples were analyzed. One was preserved with Nitric Acid, one with Hydrochloric Acid, and one as received. Each sample was analyzed along with a Matrix Spike performed on the sample. The samples preserved with hydrochloric acid did not analyze well, and results were not obtained. The other samples were consistent with the originally reported results; however, the Matrix Spike showed excellent recovery. This recovery rules out overloading of the carbon-14 trap. The Nitric Acid was also tested for the conversion of Carbon to Carbon dioxide. This follow-up test produced the same results as originally reported. The samples were also analyzed via an oil analysis method utilizing Sulfuric Acid in the beginning, then proceeding as normal. The results were consistent with the originally reported results.

GEL requested a second set of samples from DOELAP. However, the samples were never received. GEL participates in a secondary PE study via the PROCORAD organization. GEL has not had any failures under this PE study. PROCORAD utilizes real urine, while DOELAP prepares their PE samples with synthetic urine. GEL has successfully passed the PROCORAD's PE study.

Upon further investigation, it was determined the problem is not refrigerating the sample upon receipt.

This corrective action is not closed. GEL is awaiting DOELAP sample results for 2011.

Incident associated with Work Order 256295, August 11, 2010

The incident involved a lab error made by an analyst that resulted in 8 samples being reported as Failed Analysis for Strontium. Tagwords 10G0777, 10G0796, 10G0003, 10G0084, 10G0120, 10G0780, 10G0797 and 10G0800 were the affected work orders. The error occurred when running Isotopic Plutonium and Strontium sequentially on a single batch of samples. The analyst, Christina Kimball, inadvertently used centrifuge tubes labeled as Strontium to catch her Plutonium elution and vice versa. On the day of the error and before the error was noticed, she took the samples labeled Plutonium and precipitated them as Pu and then filtered. These were turned into the count room for analysis. In hindsight, these were the Strontium elutions. On the day following the error, she noticed that her samples labeled Strontium had 25 mL of liquid when they should only have 15 mL. The Group Leader was consulted. The incident was investigated. The Plutonium results were checked, as the samples had already started counting. There was no tracer yield on the samples, so, at that time, it was suspected that the Pu & Sr samples were switched. The samples labeled Strontium were precipitated as Plutonium, filtered saving the liquid residue, counted and verified Plutonium tracer peaks were present. Thus, we were able to recover and report the Plutonium fraction, but the Strontium fraction was lost in analysis due to precipitating and filtering the samples as Plutonium. In resolution, the analyst was reminded to stay focused.

This corrective action is considered closed.

Incident associated with Work Order 257200, August 17, 2010

The incident involved a batch of routine isotopic uranium samples containing work orders 257200 and 257698, which were reported as failed analysis. The affected tagwords are 10G0190, 10G0285, 10G0310, 10G0406, 10G1033, 10G1034, 10G1036, 10G1037 and 10G1038. The analyst, Christina Kimball, apparently made an error either omitting a chemical, using the wrong chemical, failing to trace & spike, failure to add Neodymium or reduce Uranium with Titanium Chloride during the final precipitation step, and etc. results in the entire batch having no tracer yields. The reason for the error is the same in all cases- due to the lack of attention to detail. In resolution, a checklist was implemented for the analysts to follow while proceeding through the complex preparation and separation procedures. The checklists are an aid to perform the steps as outlined in the Standard Operating Procedure to ensure all steps are followed as written. The analyst was re-trained and reminded to stay focused.

This corrective action is considered close.

Incident associated with Work Order 261194, October 19, 2010

The incident involved Tagwords 10I0144, 10I0145, 10I0156, 10I0196, 10I0269, 10I0274, 10I0332, 10I0409, and 10I0641 for Plutonium analysis all have failing tracer yields. The highest is 17.656% for sample 10I0196. However, overall the average is about 5%. No LCS recovery was calculated either due to the failing tracer yields. The prep analyst was consulted and did not notate any errors/issues with the batch. The entire sample was consumed for analysis, and no samples remain for re-preps. The results are reported as Failed Analysis. The strontium portion has already been completed, and all those results are within requirements. Upon further investigation and review, it was discovered through the batch checklist that we have a step that can be omitted when Am & Pu are not required e.g. uranium only analysis. This specific set of samples had only Pu & Sr. The analyst (our newest one) omitted the Americium rinse step. This results in the TiCl3 rinse which reduces the Pu and allows it to elute from the cartridge being added to a HNO3 environment vs. a HCL environment on the cartridge, which may have been responsible for the low yields. In resolution, the group leader discussed this with the analyst. This analyst had performed several Am, Pu & Sr batches over the past couple of months but had not performed Pu without Am. A training issue was discovered and was corrected.

This corrective action is considered closed.

Incident associated with Work Order 268491, January 12, 2011

This incident involved sample with tagword 10L0551 which was received and logged via normal protocol. The single sample contained 4 tagwords each getting a different in house sample ID. Sr-90 should have been analyzed sequentially with Am,Pu,Cm and U. Analyst I Chrissi Kimball has the duties of batching samples. When batching sample 10L0551, the paperwork was inadvertently not included with the paperwork for Am,Pu,Cm and U analysis. When preparing the samples, the prep analyst, Julie Williams, did not notice the sample required Sr-90 and proceeded with analysis and did not perform Sr-90 analysis on the sample. The Sr-90 fraction was lost during analysis. In resolution, the Group Leader, Bob Timm, gathered Julie, Chrissi and other analysts who analyze similar type samples and performed on the spot training discussing the error.

This corrective action is considered closed.

Incident associated with Work Order 270079, January 19, 2011

The incident involved samples with tagwords 11A0700, 10L0475, 11A0222, 11A0341, 11A0484, 11A0500, 11A0501, 11A0539 and 11A0707. The samples are in work order's 270079, 270081 and 270183. An analyst error resulted in 9 isotopic uranium samples, from a single batch, to be reported as Failed Analysis. The analyst failed to catch the Uranium fraction as they confused this analysis which was Pu/U with Am/Pu.

Upon investigation by the Group Leader Bob Timm, it was discovered that the error occurred while performing the column separations for batches 1063614 and 1063616, which were being analyzed sequentially. While preparing the chemical separations, the analyst set up the batches for Americium and Plutonium cartridges instead of the required Plutonium and Uranium cartridges. The error was not identified until the day after the separations took place and all of the waste fractions and the cartridges containing the Uranium fraction had been discarded.

In resolution, Mr. Timm reviewed this occurrence with the preparation analysts of the laboratory. He discussed the importance of a new procedural change for chemical separations. The lab analysts have been instructed to perform and document a witness evaluation just prior to chemical separations and immediately after chemical separations. Prior to the separation, a second analyst will examine the setup for separations and verify on the paperwork that all required analytes are being separated properly. This will be documented on the upper left hand corner of the laboratory batch sheet. At the end of the chemical separations and before discarding any wastes or resin cartridges, a second analyst will examine the retained fractions and verify all required analytes have been prepared. This will be documented by an additional set of initials and date on the batch checklist. Documentation of this training session is on file for each analyst in attendance.

This corrective action is considered closed.

Incident associated with Work Order 272967, March 09, 2011

The incident involved Tagword 11B0961. There was an issue with an incorrect status assignment of a sample within SDG 272967. Upon investigation, GEL's CST department audited the table that contained the data and was able to determine the sequence of events of the sample status change; however, they did not have a program in place to alert someone when a status change occurs that should not. In resolution, CST has programmed an email notification that will be sent to the Project Manager whenever a sample is status to IS (insufficient sample).

This corrective action is considered closed.

Incident associated with Work Order 273390, March 31, 2011

The incident involved samples with tagwords 11B0724, 11C0330, 11C0536, and 11C0396. A prep error occurred which resulted in four samples reported as a failed analysis for Plutonium due to low tracer yields. Analyst Dave Johnston prepared the samples. Group leader Bob Timm reviewed the data for any potential error. Mr. Johnston prepared a batch for another client before this batch, and it had a lower average tracer yield. Two other prep analysts are performing well so this does not appear to be a method problem, only the execution of the method by the analyst.

In resolution, Mr. Johnston has been removed from Plutonium in urine analysis until a remedial qualification can be performed.

This corrective action is considered closed.

Corrective Actions

There is one corrective action at this time pertaining to work order 245645.

APPENDIX C

QUALITY CONTROL INTERCOMPARISON PARTICIPATION RESULTS

(Historical File Only)

SECTION 3

DOELAP/NRIP RESULTS

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-10-SF
Test Radionuclides: ^{241}Am, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{235}U, ^{234}U, ^{90}Sr, ^{237}Np, ^{60}Co, ^{137}Cs
Matrix Description: Synthetic Feces[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, April 1, 2010

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)
^{241}Am	0.763	0.82	0.650	11.8	-14.8
^{238}Pu	0.511	0.71	0.449	19.6	-12.2
^{240}Pu	0.785	0.79	0.722	12.5	-8.11
^{230}Th	1.036	0.61	0.924	19.7	-10.8
^{238}U	0.912	0.63	0.888	12.1	-2.7
^{234}U	0.879	0.98	0.912	12.0	3.8
^{235}U	0.042	0.80	0.041	51.8	-1.9
^{90}Sr	71.5	0.77	68.0	10.3	-4.9
^{137}Cs	73.6	0.72	69.7	16.0	-5.2
^{60}Co	37.3	0.54	35.8	22.4	-3.9

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
^{241}Am	Yes	15	Pass	Pass
^{238}Pu	Yes	26	Pass	Pass
^{240}Pu	Yes	17	Pass	Pass
^{230}Th	Yes	26	Pass	Pass
^{238}U	Yes	18	Pass	Pass
^{234}U	Yes	19	Pass	Pass
^{235}U	Yes	76	Pass	Pass
^{90}Sr	Yes	15	Pass	Pass
^{137}Cs	Yes	23	Pass	Pass
^{60}Co	Yes	32	Pass	Pass

Samples Distributed August 12, 2010
Reporting Data Received October 15, 2010

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-10-SF
Test Radionuclides: ^{241}Am, ^{238}Pu, ^{240}Pu, ^{230}Th, ^{238}U, ^{235}U, ^{234}U, ^{90}Sr, ^{237}Np, ^{60}Co, ^{137}Cs
Matrix Description: Synthetic Feces[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, April 1, 2010

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)
^{241}Am	0.763	0.82	0.690	16.0	-9.4
^{238}Pu	0.511	0.71	0.467	14.3	-8.6
^{240}Pu	0.785	0.79	0.725	13.5	-7.7
^{230}Th	1.036	0.61	0.992	13.5	-4.2
^{238}U	0.912	0.63	0.890	12.4	-2.5
^{234}U	0.879	0.98	0.894	14.5	1.8
^{235}U	0.042	0.80	0.043	44.7	3.5
^{90}Sr	71.5	0.77	62.9	10.8	-12
^{137}Cs	73.6	0.72	74.5	18.1	1.2
^{60}Co	37.3	0.54	35.6	43.8	-4.5

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
^{241}Am	Yes	22	Pass	Pass
^{238}Pu	Yes	20	Pass	Pass
^{240}Pu	Yes	19	Pass	Pass
^{230}Th	Yes	19	Pass	Pass
^{238}U	Yes	18	Pass	Pass
^{234}U	Yes	22	Pass	Pass
^{235}U	Yes	69	Pass	Pass
^{90}Sr	Yes	14	Pass	Pass
^{137}Cs	Yes	28	Pass	Pass
^{60}Co	Yes	63	Pass	Pass

Samples Distributed August 12, 2010
Reporting Data Received October 15, 2010

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.

A continuing traceability program in radioactivity demonstrates, to the degree established by the periodic assays of calibrated radioactivity samples, a continuing competence to maintain the instrument systems and standards necessary for accurate measurement. Such a program cannot, however, endorse each and every measurement nor the final product, any more than a spot check can vouch for every unchecked item. Care should be taken, therefore, not to imply such endorsement. The proper use of this Report is governed by section 200.114 of Title 15 of the Code of Federal Regulations. These regulations may be met if Reports are quoted only in their entirety. Excerpts out of context may be misleading.

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 2010). 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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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: NRIP10-SU Set #1
Test Radionuclides: ^{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, gross alpha, gross beta in acidified water[1]
Matrix Description: Synthetic Urine[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, August 1, 2010

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)
^{57}Co	77.16	1.88	88	15.7	13.5
^{60}Co	911.1	0.60	942	11.0	3.4
^{90}Sr	49.97	0.79	49.9	11.6	-0.1
^{137}Cs	1028.1	0.78	1082	11.0	5.2
^{210}Po	22.47	3.23	20.9	11.2	-6.9
^{226}Ra	4.927	0.89	4.35	39.9	-11.7
^{230}Th	2.908	0.62	2.84	12.1	-2.4
^{234}U	6.139	1.00	5.56	11.3	-9.4
^{235}U	0.293	0.66	0.273	31.3	-6.9
^{238}U	6.373	0.64	5.86	15.4	-8.0
^{238}Pu	1.892	0.72	1.85	13.1	-2.0
^{240}Pu	2.398	0.79	2.30	12.6	-4.2
^{241}Am	5.623	0.83	5.51	14.4	-1.9
^{243}Cm	2.322	1.05	2.35	14.0	1.0

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha-, Beta- and Gamma-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
^{57}Co	Yes	27	Pass	Pass
^{60}Co	Yes	17.1	Pass	Pass
^{90}Sr	Yes	17.5	Pass	Pass
^{137}Cs	Yes	17.5	Pass	Pass
^{210}Po	Yes	16.4	Pass	Pass
^{226}Ra	Yes	53	Pass	Pass
^{230}Th	Yes	17.8	Pass	Pass
^{234}U	Yes	15.4	Pass	Pass
^{235}U	Yes	44	Pass	Pass
^{238}U	Yes	21.2	Pass	Pass
^{238}Pu	Yes	19.3	Pass	Pass
^{240}Pu	Yes	18.1	Pass	Pass
^{241}Am	Yes	21.2	Pass	Pass
^{243}Cm	Yes	21.2	Pass	Pass

Samples Distributed July 15, 2010
Reporting Data Received September 14, 2010

For the Director

Michael P. Unterweger,
Group Leader
Radioactivity Group
Physical Measurement Laboratory

(Continued)

Notes

(1a) Five test-samples and three (identical matrix) blanks were provided for this test. Each sample consisted of approximately 1000 or 100 grams of synthetic urine material contained in a 1-L or 125 mL size polyethylene bottle.

Composition of the Synthetic Urine

Reagent		Weight/1L Sample (g)
$H_2C_2O_4 \cdot 2H_2O$	Oxalic Acid	0.02
Pepsin	Pepsin	0.029
$CH_3CHOHCO_2H$	Lactic Acid (liquid)	0.094
$MgSO_4 \cdot 7H_2O$	Magnesium Sulfate	0.46
$C_5H_{11}O_5CHO$	Glucose (dextrose)	0.48
Citric Acid	Citric Acid	0.54
$CaCl_2 \cdot 2H_2O$	Calcium Chloride	0.63
$C_9H_9NO_3$, 98%	Hippuric Acid	0.63
$Na_2SiO_3 \cdot 9H_2O$	Sodium Silicate	0.071
NH_4Cl, 99%	Ammonium Chloride	1.06
$C_4H_9N_3O_2 \cdot H_2O$	Creatine	1.1
NaCl, 99+%	Sodium Chloride	2.32
$NaH_2PO_4 \cdot H_2O$	Sodium Dihydrogen Phosphate	2.73
KCl	Potassium Chloride	3.43
Na_2SO_4	Sodium Sulfate	4.31
CH_4N_2O, 98%	Urea	16
HNO_3	Concentrated nitric acid (50 mL)	70.7
H_2O	Water	950
Total Sample		**1054.6**

(1b) The test samples were prepared by adding a known amount of a NIST calibrated "spike" solution (aqueous solution containing 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, ^{243}Cm to the bottle with the urine matrix.

(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 ^{210}Po reference date was 12:00 EST, 26 August 2010, the date of measurement reported by the laboratory.

Note: Half-life data are based mainly on DDEP-Working Group recommended values (see References). Uncertainties are quoted at the 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)	^{57}Co	Pressurized "4π" γ ionization chamber "A" calibrated using a cobalt-57 solution whose activity was determined by "4π"-(β+γ)-coincidence and anti-coincidence counting
b)	^{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
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}Pb	4π(α+β) liquid-scintillation counting systems
f)	^{230}Th	Two 4πα liquid scintillation counting systems
g)	^{226}Ra	Pressurized "4π" γ ionization chamber "A"
h)	^{234}U, ^{235}U, ^{238}U	Mass spectrometry, silicon surface barrier alpha-detection, and 4π (α+β) liquid-scintillation counting systems
i)	^{238}Pu	Two 4πα liquid scintillation counting systems
j)	^{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
k)	^{241}Am	4πα liquid-scintillation counting system
l)	^{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.

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: NRIP10-SU Set #2
Test Radionuclides: ^{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, gross alpha, gross beta in acidified water[1]
Matrix Description: Synthetic Urine[1]
Test Activity Range: 30mBq•sample^{-1} to 300mBq•sample^{-1}
Reference Time: 12:00 EST, August 1, 2010

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)
^{57}Co	77.16	1.88	86	25.6	11.0
^{60}Co	911.1	0.60	932	11.1	2.3
^{90}Sr	49.97	0.79	48.7	11.6	-2.5
^{137}Cs	1028.1	0.78	1088	11.0	5.8
^{210}Po	22.47	3.23	23.0	11.2	2.2
^{226}Ra	4.927	0.89	4.48	48.3	-9.1
^{230}Th	2.908	0.62	2.76	12.2	-5.0
^{234}U	6.139	1.00	6.17	21.3	0.5
^{235}U	0.293	0.66	0.312	41.6	6.3
^{238}U	6.373	0.64	6.21	29.1	-2.6
^{238}Pu	1.892	0.72	1.80	13.8	-4.8
^{240}Pu	2.398	0.79	2.23	13.1	-7.2
^{241}Am	5.623	0.83	5.18	14.3	-7.9
^{243}Cm	2.322	1.05	2.04	17.7	-12.3

Methods

	NIST[6]	Reporting Laboratory[7]
Activity Measurements	Alpha-, Beta- and Gamma-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
^{57}Co	Yes	43	Pass	Pass
^{60}Co	Yes	17.0	Pass	Pass
^{90}Sr	Yes	16.9	Pass	Pass
^{137}Cs	Yes	17.5	Pass	Pass
^{210}Po	Yes	17.8	Pass	Pass
^{226}Ra	Yes	66	Pass	Pass
^{230}Th	Yes	17.5	Pass	Pass
^{234}U	Yes	32	Pass	Pass
^{235}U	Yes	66	Pass	Pass
^{238}U	Yes	42	Pass	Pass
^{238}Pu	Yes	19.7	Pass	Pass
^{240}Pu	Yes	18.3	Pass	Pass
^{241}Am	Yes	19.8	Pass	Pass
^{243}Cm	Yes	23	Pass	Pass

Samples Distributed July 15, 2010
Reporting Data Received September 14, 2010

For the Director

Michael P. Unterweger,
Group Leader
Radioactivity Group
Physical Measurement Laboratory

(Continued)

Notes

(1a) Five test-samples and three (identical matrix) blanks were provided for this test. Each sample consisted of approximately 1000 or 100 grams of synthetic urine material contained in a 1-L or 125 mL size polyethylene bottle.

Composition of the Synthetic Urine

Reagent		Weight/1L Sample (g)
$H_2C_2O_4 \cdot 2H_2O$	Oxalic Acid	0.02
Pepsin	Pepsin	0.029
$CH_3CHOHCO_2H$	Lactic Acid (liquid)	0.094
$MgSO_4 \cdot 7H_2O$	Magnesium Sulfate	0.46
$C_5H_{11}O_5CHO$	Glucose (dextrose)	0.48
Citric Acid	Citric Acid	0.54
$CaCl_2 \cdot 2H_2O$	Calcium Chloride	0.63
$C_9H_9NO_3$, 98%	Hippuric Acid	0.63
$Na_2SiO_3 \cdot 9H_2O$	Sodium Silicate	0.071
NH_4Cl, 99%	Ammonium Chloride	1.06
$C_4H_9N_3O_2 \cdot H_2O$	Creatine	1.1
NaCl, 99+%	Sodium Chloride	2.32
$NaH_2PO_4 \cdot H_2O$	Sodium Dihydrogen Phosphate	2.73
KCl	Potassium Chloride	3.43
Na_2SO_4	Sodium Sulfate	4.31
CH_4N_2O, 98%	Urea	16
HNO_3	Concentrated nitric acid (50 mL)	70.7
H_2O	Water	950
Total Sample		**1054.6**

(1b) The test samples were prepared by adding a known amount of a NIST calibrated "spike" solution (aqueous solution containing 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, ^{243}Cm to the bottle with the urine matrix.

(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 ^{210}Po reference date was 12:00 EST, 26 August 2010, the date of measurement reported by the laboratory.

Note: Half-life data are based mainly on DDEP-Working Group recommended values (see References). Uncertainties are quoted at the 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)	^{57}Co	Pressurized "4π" γ ionization chamber "A" calibrated using a cobalt-57 solution whose activity was determined by "4π"-(β+γ)-coincidence and anti-coincidence counting
b)	^{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
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}Pb	4π(α+β) liquid-scintillation counting systems
f)	^{230}Th	Two 4πα liquid scintillation counting systems
g)	^{226}Ra	Pressurized "4π" γ ionization chamber "A"
h)	^{234}U, ^{235}U, ^{238}U	Mass spectrometry, silicon surface barrier alpha-detection, and 4π (α+β) liquid-scintillation counting systems
i)	^{238}Pu	Two 4πα liquid scintillation counting systems
j)	^{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
k)	^{241}Am	4πα liquid-scintillation counting system
l)	^{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.

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Performance Evaluation Results

Session 13

Synthetic Urine / Synthetic Fecal

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Performance Evaluation Results

Laboratory Summary Reports

DEPARTMENT OF ENERGY
LABORATORY ACCREDITATION PROGRAM

Summary Report

Laboratory: LAB 2 Session: 0110

Matrix: SF

RESL LogNo	Radionuclide	Mean Bias (Br):	St. Dev Bias (Sb)	Pass / Fail
DL423	Am-241	-0.015	0.028	Pass
DL423	Co-60	0.072	0.067	Pass
DL423	Cs-137	0.045	0.052	Pass
DL423	Pu-238	-0.046	0.039	Pass
DL423	Pu-239	-0.059	0.055	Pass
DL423	Sr-90	-0.077	0.035	Pass
DL423	U-234	-0.074	0.080	Pass
DL423	U-238	-0.062	0.056	Pass
DL424	Np-237	-0.035	0.033	Pass
DL424	Th-228	-0.067	0.039	Pass
DL424	Th-230	-0.015	0.069	Pass
DL424	Th-232	-0.114	0.033	Pass

Acceptance Criteria: -0.25 =< Br <= 0.50 Sb =< 0.4

RADIOLOGICAL AND ENVIRONMENTAL SCIENCES LABORATORY

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Summary Report

Laboratory: LAB 2 Session: 0110

Matrix: SU

RESL LogNo		Radionuclide	Mean Bias (Br):	St. Dev Bias (Sb)	Pass / Fail
DL384		C-14	-0.588	0.064	Fail
DL384		H-3	0.105	0.044	Pass
DL388		Am-241	-0.061	0.036	Pass
DL388		Co-60	0.137	0.162	Pass
DL388		Cs-137	-0.009	0.057	Pass
DL388		Pu-238	-0.104	0.081	Pass
DL388		Pu-239	-0.066	0.094	Pass
DL388		Sr-90	-0.055	0.065	Pass
DL388		U-234	-0.043	0.065	Pass
DL388		U-238	-0.053	0.051	Pass
DL389		Np-237	-0.037	0.058	Pass
DL389		Th-228	0.148	0.039	Pass
DL389		Th-230	0.119	0.079	Pass
DL389		Th-232	0.011	0.071	Pass
DL416	*	U-238	-0.054	0.036	Pass
DL416	*	U-Tot	-0.104	0.025	Pass

Acceptance Criteria: -0.25 =< Br <= 0.50 Sb =< 0.4

* Mass Determination

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