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RESULTS OF THE EXCRETA BIOASSAY QUALITY CONTROL PROGRAM FOR APRIL 1, 2008 THROUGH MARCH 31, 2009

CL Antonio

June 2010

Pacific Northwest
NATIONAL LABORATORY

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RESULTS OF THE EXCRETA BIOASSAY QUALITY CONTROL PROGRAM FOR APRIL 1, 2008 THROUGH MARCH 31, 2009

Cheryl L. Antonio

May 2010

Peer Reviewed by _____
Jay A. MacLellan Date

SUMMARY

A total of 62 urine samples and 6 spiked fecal samples were submitted during the report period (April 1, 2008 through March 31, 2009) to General Engineering Laboratories, South Carolina by the Hanford Internal Dosimetry Program (IDP) to check the accuracy, precision, and detection levels of their analyses. Urine analyses for Sr, ^{238}Pu, ^{239}Pu, ^{241}Am, ^{243}Am ^{235}U, ^{238}U, elemental uranium and fecal analyses for ^{241}Am, ^{238}Pu and ^{239}Pu were tested this year. The number of QC urine samples submitted during the report period represented 1.3% of the total samples submitted.

In addition to the samples provided by IDP, GEL was also required to conduct their own QC program, and submit the results of analyses to IDP. About 34% of the analyses processed by GEL during the third year of this contract were quality control samples. GEL tested the performance of 21 radioisotopes, all of which met or exceeded the specifications in the Statement of Work within statistical uncertainty (Table 4).

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

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

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, ^{233,234}U was not evaluated. 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 elemental uranium urinalysis program and it was considered acceptable. Because IDP uses a 0.2 µg screening level for elemental uranium, samples spiked at 0.06 µg were discontinued. The MDA at the contractual level of 0.06 µg was evaluated through GEL's program and was found to be acceptable. The relative bias and precision were likewise acceptable. The bias and precision 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/L and at 0.05/L µg.

The total strontium procedure is used to screen samples to determine which will require analysis for ^{90}Sr. Samples with total strontium results less than 15 dpm do not undergo further analysis. Samples with results greater than or equal to 15 dpm may undergo ^{90}Y in growth to specifically determine ^{90}Sr levels. The calculated MDA, reported by GEL and tested by IDP, for the total strontium part of the analysis was less than 30% of the CL. The relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all within limits. The 19 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 20 samples spiked at the CL for ^{239}Pu were reported with only one result less than the decision level (i.e., not detected). There were 25 blank samples analyzed for ^{238}Pu activity, none of the 25 samples detected activity in excess of the decision level. Overall the plutonium urinalyses were considered acceptable.

The MDA and performance statistics for ^{239}Pu and ^{238}Pu in feces were acceptable. Approximately 15% of the fecal samples analyzed were duplicated to test the consistency of the aliquoting procedure. A review of the duplicate samples determined that the aliquoting procedure produced results within 3 sigma of the initial results. The fecal aliquoting procedure was acceptable. This year IDP submitted 6 actual fecal samples spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The precision and bias for ^{239}Pu and the relative bias for ^{238}Pu met the performance criteria. The relative precision for ^{238}Pu slightly exceeded the criteria. The performance statistics reported by GEL for ^{239}Pu met the acceptance criterion; however, GEL did not test ^{238}Pu. The relative bias and precision for ^{238}Pu will be reviewed again in the 2009 through 2010 contract year. The failed analysis rate for fecal sampling was 19%, which exceeded the contractual level of 10%. The problem appears to be technician errors. Overall the plutonium fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

The ^{241}Am fecal and urine analysis met the acceptance criteria for MDA, relative bias and precision. The MDA as reported by GEL and tested by IDP was less than 50% of the contractual level. Five blank samples submitted by IDP in August 2008 had elevated ^{241}Am activity because the spike source was contaminated; the five samples were not included in the MDA calculation. A more detailed discussion of the cross contamination is in the ^{241}Am discussion section. All 14 of the ^{241}Am samples spiked at the contractual detection level (CL) were detected. The relative bias and precision as reported by GEL and tested by IDP met the performance criteria. The current AM241 urinalysis procedure was considered acceptable.

The ^{241}Am fecal duplicate samples were evaluated and it was concluded that the aliquoting procedure produced results within the control limits. This year IDP submitted 6 actual fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. The failed analysis rate for ^{241}Am fecal analyses was 20%, which exceeded the contractual level of 10%. The problem appears to be technician errors. Overall the ^{241}Am fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

The ^{243}Am procedure was identical to the ^{241}Am 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 ^{243}Am procedure was concluded to be acceptable.

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

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

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

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Tables

INTRODUCTION

This report summarizes the results of the excreta bioassay quality control program's monitoring of the performance of General Engineering Laboratories (GEL) for samples submitted from April 1, 2008 through March 31, 2009. During the reporting period GEL analyzed, under the contract with Battelle, 4980 urine and 42 fecal samples for various radionuclides. This is about the same workload as reported in the 2007 report.

The results of the analyses are part of a system of legal records concerning internal deposition of radionuclides for workers at the Hanford Site. GEL is required to have a rigorous quality control (QC) program to ensure the accuracy of its results. In addition, the Pacific Northwest National Laboratory's (PNNL) Hanford Internal Dosimetry Program (IDP) has a QC program in place to independently check the accuracy of the results from GEL. The objective of the PNNL excreta bioassay QC program is to provide quantitative data to support the assessment of performance criteria for excreta bioassay analyses, as specified in the Statement of Work (Battelle 2007).

The reliability of the excreta bioassay program depends, to a significant extent, on the adoption and implementation of performance criteria for laboratory accuracy, precision, and detection levels.

Such performance criteria are established in the Statement of Work (Battelle 2007) and include the following:

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

Formulas for MDA, B_r, and S_B, presented in the next section of this report, are based on recommendations in the Health Physics Society (HPS) Standard N13.30 (1996) and are listed in the Statement of Work (SOW). In addition to the 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).

GENERAL METHODS

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

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

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

Battelle Contract 11530 - Feb-06

Table B-3

TABLE 1. Analytical and Reporting Requirements for Routine Processing of Samples

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

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

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

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

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

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

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

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

Table B-3: Effective 1/7/2009

TABLE 1. Analytical and Reporting Requirements for Routine Processing of Samples (continued)

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

	THIRD CONTRACT YEAR - GEL				FOURTH CONTRACT YEAR - GEL			
Procedure	**4/1/07 through 3/31/08**				**4/1/08 through 3/31/09**			
Code[a]	Total	IDP QC	%	GEL QC[b]	Total	IDP QC	%	GEL QC[b]
Urine								
H3	821	0	0.0	282	664	0	--	178
SR90, SR	181	0	0.0	447	282	0	--	522
C14	--	--	--	--	--	0	--	--
AM241	99	--	--	463	96	0	--	402
AM243	88	7	8.0	84	70	7	10	66
U235	--	--	--	--	--	0	--	--
ICM	7	--	--		8	0	--	
IPU	1401	9	--	1261	1406	0	--	1065
IPUL	5	--	--	--	2	0	--	--
IPA	401	4	1.0	N/A	445	0	--	N/A
IPS	481	0	0.0	N/A	722	0	--	N/A
IPSA	158	20	12.7	N/A	172	25	15	N/A
IPSR	--	--	--	--	--	0	--	--
ISPEC	--	--	--	--	--	0	--	--
ITPAC	116	--	--	N/A	220	0	--	N/A
ITH	1	--	--	8	3	0	--	6
IUPU	114	--	--	N/A	93	0	--	N/A
IPIU	10	0	0.0	N/A	10	0	--	N/A
IU	519	16	3.1	243	467	9	2	189
NP237	--	--	--	--	7	0	--	--
U236	1	--	--	3	20	15	75	8
UNAT	218	23	10.6	462	293	6	2	180
LEPD	--	--	--	--	--	0	--	--
PU241	--	--	--	--	--	0	--	--
Total	***4621***	***79***	***1.7***	***3253***	***4980***	***62***	***1***	***2616***
Fecal [c]								
U232	--	--	--	--	--	0	--	--
ICM	--	--	--	--	--	0	--	--
IU	3	1	--	7	--	0	--	--
AM241	4	--	--	86	--	0	--	79
IPU	36	--	--	116	1	0	--	82
IPA	55	7	12.7	N/A	41	6	15	N/A
Total	***98***	***8***	***8.2***	***209***	***42***	***6***	***14***	***161***

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

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

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

Table 2 presents a breakdown of the numbers and categories for all bioassay samples analyzed, including personnel and QC samples. From 62 urine and 6 fecal QC samples submitted by IDP to GEL during the reporting period, GEL reported 4980 analytical urine results for 13 different analytes and 42 fecal results for 3 different analytes. The 68 QC samples represent 1.3% of the total analyses performed by GEL. In addition to these samples, GEL analyzed 2777 internal QC samples. The QC samples analyzed equaled 34% 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:

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

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.

BIAS

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

The mean relative bias, Br, is determined using:

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

where n = number of spike samples in each level
m = number of spike levels
N = total number of spiked samples

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

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

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

A_{ai} = the true value of the ith spike level

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

Matrix	Nuclide/ Method	Count Minutes	*Contract Limit*[(a)]	Counter Efficiency 2007-2008	Counter Efficiency 2008-2009	Chemical Yield 2007-2008	Chemical Yield 2008-2009
Urine	^{3}H	20	*20*	0.24	0.243	---	
	Total Sr	60	*10*	0.379	0.379	0.788	0.757
	SR90	60	*10*	---	0.379	---	0.76
	^{241}Am	2520	*0.02*	0.391	0.391	0.816	0.8175
	^{243}Am	2520	*0.02*	0.391	0.391	0.871	0.8668
	$^{242}Cm/^{244}Cm$	2520	*0.02*	0.391	0.391	0.816	0.8175
	^{237}Np	2520	*0.02*	---	---	---	---
	$^{239}Pu/^{238}Pu$	2520	*0.02*	0.391	0.391	0.890	0.925
	IPUL	10000	*0.005*	---	---	---	---
	$^{228}Th/^{230}Th/^{232}Th$	2520	*0.1*	0.386	0.386	0.880	0.9156
	$^{234}U/^{235}U/^{238}U$	2520	*0.02*	0.386	0.386	0.834	0.9047
	Uranium	--	*0.06*	N/A	N/A	N/A	N/A
Fecal	^{241}Am	960	*0.8*	0.391	0.391	0.757	0.909
	$^{238}Pu/^{239}Pu$	960	*0.2*	0.391	0.391	0.85	0.914

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

(b) Only one sample analyzed

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

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

PRECISION

The precision statistic used for this contract was S_B from HPS N13.30 (1996), but the limits differ from that standard. S_B is given by:

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

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 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) did not identify any outliers. However, there were 14 analytical ^{241}Am urinalysis results spiked at the CL that were determined to be outliers. These samples indicated activity between three to five times the CL. An investigation concluded that the samples were contaminated in the RPG laboratory during spiking. All 14 urine samples were spiked with 0.02 dpm ^{239}Pu and 0.02 dpm ^{241}Am, unfortunately the ^{239}Pu source material was contaminated with ^{241}Am. The 14 data points were subsequently removed from the data set.

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	113	0.6376	0.882	**20**	113	0.002	0.07	0	…	…
Total Sr	**IDP**	6	1.06	2.35	**10**	19	-0.062	0.11	0	…	…
	GEL	58	0.26	2.81	**10**	58	-0.01	0.11	58	0.01	0.06
^{90}Sr	GEL	165	0.55	3.32	**10**	164	0.002	0.12	166	0.012	0.084
^{228}Th	GEL	3	0.006	0.009	**0.1**	0	…	…	0	…	…
^{229}Th	GEL	3	0.010	0.019	**0.1**	0	…	…	0	…	…
^{232}Th	GEL	3	0.008	0.015	**0.1**	3	0.02	0.10	3	0.037	0.01
^{230}Th	GEL	3	0.021	0.034	**0.1**	0	…	…	0	…	…
^{242}Cm	GEL	48	0.004	0.011	**0.02**	0	…	…	0	…	…
^{243,244}Cm	GEL	173	0.004	0.011	**0.02**	48	0.04	0.38	48	0.048	0.115
^{238}Pu-urine	**IDP**	25	0.003	0.009	**0.02**	0	…	…	0	…	…
	GEL	454	0.004	0.009	**0.02**	0	…	…	0	…	…
feces	**IDP**	0	…	…	**0.2**	6	0.13	0.5044[c.]	0	…	…
	GEL	21	0.01	0.060	**0.2**	0	…	…	0	…	…
^{239,240}Pu-urine	**IDP**	5	0.006	0.016	**0.02**	20	-0.04	0.32	0	…	…
	GEL	454	0.004	0.010	**0.02**	452	0.06	0.28	456	0.015	0.071
feces	**IDP**	0	…	…	**0.2**	6	-0.04	0.16	0	…	…
	GEL	21	0.07	0.014	**0.2**	21	-0.02	0.22	21	-0.001	0.052
^{241}Am-urine	**IDP**	6	0.0014	0.0078	**0.02**	14	0.18	0.32	0	…	…
	GEL	173	0.005	0.011	**0.02**	168	0.11[e]	0.27	177	0.045	0.108
feces	**IDP**	0	…	…	**0.2**	6	-0.03	0.06	0	…	…
	GEL	20	0.01	0.047	**0.2**	20	0.03	0.14	18	0.021	0.078
^{243}Am-urine	**IDP**	7	0.005	0.015	**0.02**	0	…	…	0	…	…
	GEL	26	0.006	0.012	**0.02**	0	…	…	0	…	…
^{233,234}U	**IDP**	0	…	…	**0.02**	0	…	…	0	…	…
	GEL	81	0.008	0.017	**0.02**	0	…	…	0	…	…
^{235,236}U	**IDP**	9	N/A	0.014	**0.02**	0	…	…	0	…	…
	GEL	81	0.006	0.014	**0.02**	0	…	…	0	…	…
^{238}U	**IDP**	0	…	…	**0.02**	0	…	…	9	-0.07	0.08
	GEL	81	0.007	0.016	**0.02**	81	-0.11[e]	0.31	81	-0.121[e]	0.12
^{236}U (ICPMS)[b]	**IDP**	10	114 [c]	229 [c]	**140 pg**	5	-0.11	0.07	0		
	GEL	4	12.5	40.4	**140 pg**	0	…	…	4.00	-0.02	0.03
U-urine [b]	**IDP**	0	…	…	**0.06 µg**	0	…	…	6	-0.19	0.25
	GEL	140	0.006	0.01	**0.06 µg**	47	-0.20	0.22	47	-0.180	0.137

(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	2008	0	…	…	0	…	…	0	…	…
		2007	0	…	…	0	…	…	0	…	…
Sr	10 dpm	2008	6	1.06	2.35	19	-0.06	0.11	0	…	…
		2007	0	…	…	20	0.003	0.095	0	…	…
U	0.06 mg	2008	0	…	…	0	…	…	6	-0.19	0.25
(elemental)		2007	0	…	…	0	…	…	22	-0.06	0.32
^{235}U	0.02 dpm	2008	9	N/A	0.01	0	…	…	0	…	…
		2007	8	…	0.0197	0	…	…	0	…	…
^{238}U	0.02 dpm	2008	0	…	…	0	…	…	9	-0.07	0.08
		2007	0	…	…	0	…	…	16	-0.02	0.30
^{238}Pu	0.02 dpm	2008	25	0.003	0.009	0	…	…	0	…	…
(urine)		2007	32	0.011	0.025[e]	0	…	…	0	…	…
^{238}Pu	0.2 dpm	2008	0	…	…	6	0.13	0.504(c.)	0	…	…
(fecal)		2007									
^{239}Pu	0.02 dpm	2008	5	0.006	0.016	20	-0.04	0.32	0	…	…
(urine)		2007	0	…	…	33	-0.02	0.30	0	…	…
^{239}Pu	0.2 dpm	2008	0	…	…	6	-0.04	0.16	0	…	…
(fecal)		2007	2	0.04	0.20	5	-0.10	0.11	0	0	0
^{241}Am	0.02 dpm	2008	6	0.0014	0.0078	14	0.18	0.32	0	…	…
(urine)		2007	0	…	…	25	0.14	0.50	0	…	…
^{241}Am	0.2 dpm	2008	0	…	…	6	-0.03	0.06	0	…	…
(fecal)		2007	2	0.03	0.21	5.0	-0.08	0.10	0	…	…
^{243}Am	0.02 dpm	2008	7	0.005	0.015	0	…	…	0	…	…
		2007	7	0.006	0.016	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)

			Performance Evaluation Samples				Analytical Samples	
			Spikes at CDL		False Negatives (%)		2008-2009	
	IDP QC Samples						Yield	Failed
Nuclide	Analyses	Outliers	IDP	GEL	IDP	GEL	Flags	Analyses
Urine								
^{3}H	0	0 (0)	0	113		0 (0)		
Sr	25	0 (0)	19	58	0 (0)	0 (0)	1.7%	0.8%
^{235}U	9	0 (0)	0	0			3.8%	0.5%
^{238}U	18	0 (0)	0	81	0 (0)	0 (0)	3.8%	0.5%
^{238}Pu	25	0 (0)	0	0				1.4%
^{239}Pu	25	0 (0)	20	452	1 (5%)	0 (0)	2.2%	1.4%
^{241}Am	25	5 (20%)	14	168	0 (0)	0 (0)	1.5%	1.2%
^{243}Am	7	0 (0)	0	0				
Unat	6	0 (0)	0	47		0 (0)		0.3%
Total	***140***		***53***	***919***				
Feces								
^{241}Am	6	0 (0)	6	20	0 (0)	0 (0)		20%
^{238}Pu	6	0 (0)	6	0	0 (0)		13%	19%
^{239}Pu	6	0 (0)	6	21	0 (0)	0 (0)	13%	19%
Total	***18***		***18***	***41***				

TRITIUM

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

STRONTIUM-90 AND TOTAL STRONTIUM

The total strontium procedure is used to screen samples to determine which will require analysis for ^{90}Sr. Samples with total strontium results less than 15 dpm do not undergo further analysis. Samples with results greater than or equal to 15 dpm may undergo ^{90}Y in growth to specifically determine ^{90}Sr levels. The calculated MDA, reported by GEL and tested by IDP, for the total strontium part of the analysis was less than 30% of the CL. The relative bias and precision, tested by IDP and GEL for the ^{90}Sr and total Sr procedures were all within limits. The 19 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 20 samples spiked at the CL for ^{239}Pu were reported with only one result less than the decision level (i.e., not detected). There were 25 blank samples analyzed for ^{238}Pu activity, none of the 25 samples detected activity in excess of the decision level. Overall the plutonium urinalyses were considered acceptable.

The MDA and performance statistics for ^{239}Pu and ^{238}Pu in feces were acceptable. Approximately 15% of the fecal samples analyzed were duplicated to test the consistency of the aliquoting procedure. A review of the duplicate samples determined that the aliquoting procedure produced results within 3 sigma of the initial results. The fecal aliquoting procedure was acceptable. This year IDP submitted 6 actual fecal samples spiked with very insoluble ^{239}Pu and slightly soluble ^{238}Pu. The precision and bias for ^{239}Pu and the relative bias for ^{238}Pu met the performance criteria. The relative precision for ^{238}Pu slightly exceeded the criteria. The performance statistics reported by GEL for ^{239}Pu met the acceptance criterion; however, GEL did not test ^{238}Pu. The relative bias and precision for ^{238}Pu will be reviewed again in the 2009 through 2010 contract year. The failed analysis rate for fecal sampling was 19%, which exceeded the contractual level of 10%. The problem appears to be technician errors. 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; the relative bias reported by GEL was within statistical uncertainty and determined to be acceptable.

Because IDP used a depleted uranium source material for the isotopic uranium urinalyses, ^{233,234}U was not evaluated. 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 elemental uranium urinalysis program and it was considered acceptable. Because IDP uses a 0.2 µg screening level for elemental uranium, samples spiked at 0.06 µg were discontinued. The MDA at the contractual level of 0.06 µg was evaluated through GEL's program and was found to be acceptable. The relative bias and precision were likewise acceptable. The bias and precision 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/L and at 0.05/L µg.

Beginning next quarter, the KPA uranium mass analysis will be replaced with ICPMS analysis for ^{238}U, which comprises 99% of the uranium isotopic mixture by mass.

URANIUM-236 via Inductively Coupled Plasma Mass Spectrometry (ICPMS)

A new ^{236}U analysis procedure was initiated in June 2007 and five urinalyses were run. The analysis for ^{236}U uses an inductively coupled plasma mass spectrometry. In June 2008, IDP submitted ten blank samples and one of the 10 samples was more than 30 times greater than the average of the other nine samples. Initially it was considered an outlier; however, the sample results were valid and were therefore included in the MDA evaluation. The MDA with all 10 sample results was 229 pg, which exceeded the criteria of 140 pg. Removing the high background sample resulted in an MDA of 23 pg, which was consistent with the MDA reported by GEL. IDP also submitted five samples spiked at the CL; all five were detected and the bias and precision met the performance criteria. The procedure was formally approved in June 2008. The MDA and relative bias and precision reported by GEL met the performance criteria. The ^{236}U analysis procedure was considered acceptable.

AMERICIUM-241

The ^{241}Am fecal and urine analysis met the acceptance criteria for MDA, relative bias and precision. With regards to the urine sampling program, five blank samples submitted by IDP in August 2008 had elevated ^{241}Am activity at three-times the MDA. The five samples were part of an ongoing investigation from November 2007 into elevated ^{241}Am activity in samples spiked by the RPG laboratory. In 2007 it was determined that the contamination was not occurring at the Analytical Lab but rather in the laboratory used to spike the QC samples. The RPG laboratory investigated potential contaminated equipment, laboratory rooms and the ^{241}Am source material. As part of the investigation, five samples were spiked with ^{239}Pu but not ^{241}Am. Normally, samples spiked with plutonium-239 were likewise spiked with ^{241}Am. The five blank ^{241}Am samples had levels of ^{241}Am at three-times the MDA. Analysis of the ^{239}Pu source material detected ^{241}Am. A new ^{239}Pu source material was obtained and in August 2008 a series of ten samples consisting of blanks and spikes were tested. The sample results were valid and included in the test samples for MDA, relative bias and precision. The five blank samples from June were not included in the MDA determination as they were established to be contaminated.

The MDA for urinalyses as reported by GEL and tested by IDP was less than 50% of the contractual level. All 14 of the ^{241}Am urine samples spiked at the contractual detection level (CL) were detected. The relative bias and precision as reported by GEL and tested by IDP met the performance criteria. The current AM241 urinalysis procedure was considered acceptable.

The ^{241}Am fecal duplicate samples were evaluated and it was concluded that the aliquoting procedure produced results within the control limits. This year IDP submitted 6 actual fecal samples spiked with very insoluble ^{241}Am and the relative bias and precision were acceptable. The failed analysis rate for ^{241}Am fecal analyses was 20%, which exceeded the contractual level of 10%. The problem appears to be technician errors. Overall the ^{241}Am fecal analyses were considered acceptable but the failed analysis rate will continue to be monitored.

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 results met the acceptance criteria and the isotopic curium urinalysis program was considered acceptable.

ISOTOPIC THORIUM

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

FOLLOW-UP ON CONCERNS DURING THE FOURTH CONTRACT YEAR

The main emphasis during the first part of the fourth contract year was developing an ICPMS procedure for ^{236}U analysis. This was accomplished in June 2008. There were a few concerns carried over from the third contract year, primarily technician errors involving sample batches, typically consisting of loss of sample, cross contamination, forgetting to perform a task, or lack of proper documentation. In the first quarter there was 1 event resulting in a sample loss and three events resulting in the loss of entire sample batches by the same technician. From the second quarter on, there were no new incidents with the technician, the corrective action is considered closed. However, there continues to be sample losses due to laboratory errors, but it is not considered a serious problem. During the fourth contract year, laboratory errors resulted in 40 samples whose results were not reported, this equates to less than 1% of the total samples.

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 11530 FOURTH YEAR 2006/2007

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

In the review GEL indentified laboratory control samples that had yields greater than 125% as well as one excreta sample that had a tracer yield greater than 125%. GEL also indentified laboratory control samples that met the criteria for low yield, but likewise a review of excreta sample results found the low yield rate to be acceptable. A review by IDP of the yield rate and failed analysis rate, however, identified that 20% of the ^{241}Am fecal results were reported as failed analyses and 19% of the ^{239}Pu and ^{238}Pu. The majority of the fecal failed analyses for ^{241}Am were due to a sample batch involving cross contamination in the laboratory and were not a result of low tracer yield. Of the ^{239}Pu and ^{238}Pu fecal results 13% were reported as low-yields. This exceeded the 10% rate in the statement of work and the low yield rate will continue to be monitored through the next contract year. The urine sampling program showed acceptable levels for low-yields for all analyses. The isotopic uranium urinalysis showed the highest low yield rate at 3.8%. This was an improvement from the third contract year where the isotopic uranium low-yield rate was 13.5%.

RESULTS FROM INTERCOMPARISON PROGRAMS

GEL participated in two intercomparison programs (Appendix C – Intercomparison Programs) in the fourth contract year. Between August and October 2008, GEL participated in the National Institute of Standards and Technology's program testing the relative bias and precision for ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{243}Cm,^{238}Pu, ^{239}Pu, ^{241}Am, ^{230}Th, ^{235}U, ^{238}U, ^{234}U and ^{90}Sr in synthetic feces. GEL met the acceptance criteria for relative bias and precision for all isotopes except. GEL also participated in the National Institute of Standards and Technology's program testing the relative bias and precision for ^{241}AM+^{243}CM, ^{60}Co, ^{57}Co, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{238}Pu, ^{240}Pu, ^{241}Am, ^{230}Th, ^{235}U, ^{238}U, ^{234}U and ^{90}Sr, in synthetic urine. GEL met the acceptance criteria for relative bias and precision on all isotopes.

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RELEVANT PROCEDURES AND CORRESPONDENCE

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APPENDIX A

QUALITY CONTROL SAMPLE RESULTS
(Historical File Only)

APPENDIX B

GEL QUALITY CONTROL SAMPLE REPORT SUMMARY
(Historical File Only)

APPENDIX C

QUALITY CONTROL INTERCOMPARISON PARTICIPATION RESULTS

(Historical File Only)