April 4, 2011

Mr. Steve Tarlton, Manager
Radiation Control Program
Hazardous Materials and Waste Management Division
Colorado Department of Public Health and Environment
4300 Cherry Creek Drive South
Denver, Colorado 80246-1530

Re: Semiannual Performance Report-Laboratory and Quality Assurance Programs

Dear Mr. Tarlton,

Please find enclosed the Semiannual Performance Report-Laboratory and Quality Assurance Programs for the July - December 2010.

If you have any questions, please contact me.

Sincerely,

Jim Cain
Environmental Coordinator/
Radiation Safety Officer

JC: fwa

Attachments

PERFORMANCE REPORT

Quality Assurance Program - July - December 2010

The following discussion is prepared in accordance with the requirements for a Semiannual Performance Report on the Quality Assurance Program. This performance report evaluates data used for the Semiannual Effluent Report, the Third and Fourth Quarter 2010 Routine Water Reports, and the Third and Fourth Quarter 2010 Employee Dose Reports. The Quality Assurance Program Performance Report addresses validation of quality control samples and the data review process includes quality assurance verification and assessment of sample data. The entire data review and assessment process is addressed in this performance report.

Assessment of Cotter generated data consists of the following activities:

- Review of field data and sampling practice by sampling and quality assurance personnel.
- Review of Sample Submittal Tracking Forms (SSTF), chain of custody, and LIMS log-in documents by quality assurance personnel.
- Review and approval of the samples and QC sample data on a sample-batch basis by laboratory personnel.
- Review and approval of sample results and QC data on a sample-batch or LIMS "WorkSheet" basis by quality assurance personnel.
- Investigation of questionable or non-compliant data and subsequent follow-up.
- End-user ("Data User") data review.
- Monitoring and reporting of QC related performance criteria by quality assurance personnel.
- Evaluation of QC sample trend charts by quality assurance personnel.

Cotter assessment of reported data generated by non-Cotter offsite laboratories consists of similar activities:

- Review of Sample Submittal Tracking Forms (SSTF), chain of custody, and LIMS log-in documents by quality assurance personnel.
- Review and approval of sample results and QC data on a sample-batch or LIMS "WorkSheet" basis by quality assurance personnel.
- Investigation of questionable or non-compliant data and subsequent follow-up.
- End-user ("Data User") data review.
- Monitoring and reporting of QC related performance criteria by quality assurance personnel.
- Evaluation of QC sample trend charts by quality assurance personnel.

During any of these activities, corrective action follow-up may entail undocumented immediate request for needed investigation or corrections or the formally documented Data Request (DR), Data Verification Request (DVR) or Corrective Action – Improvement Request (CAIR) processes.

QUALITY CONTROL VALIDATION -

The May 22, 2009 Revision of the *Quality Assurance Program Plan for Environmental and Occupational Sampling and Monitoring Studies for the Cotter Corporation, Canon City Milling Facility And Lincoln Park, Colorado Superfund Site* or "QAPP Manual" established Program Performance Criteria which specify quality control and quality assurance requirements. The Program Performance Criteria require quality control data processing and data qualification or flagging. Specific to this report the Program Performance Criteria require: 1) Calculation and monitoring of the "Total Uncertainty" (TU) of accuracy (Matrix Spike) and precision (Laboratory Duplicate) sample results; 2) Determination and monitoring of batch sample result "Usability" based on TU and/or related quality control factors; 3) Monitoring the percent of qualified or "flagged" data on sample batch basis; 4) Calculation and monitoring of data "Completeness"; and 5) Monitoring and control of specific analyte detection limits.

To accomplish these requirements quality control validation begins with review of the analytical records documented in the "WorkSheet" (sample batch) data packet. Packet contents are inventoried and any batch specific notations are reviewed. Sample results and detection limits are evaluated. Quality control calculations are validated. Quality control results are evaluated for acceptability or requirement for qualification indicator ("flagging"). The WorkSheet's per cent Total Uncertainty value, %TU, is calculated and recorded. Sample batch usability is determined and recorded. From these data packet reviews, WorkSheets containing qualified data are tracked and the percent of data completeness is determined.

The implementation of all planned automated QA/QC functions relies on a fully functional Laboratory Information Management System or "LIMS". The Cotter LIMS is being upgraded to accomplish this. In the interim the %TU, usability, data qualification/flagging and completeness are being determined and recorded by the data reviewer. The Quality Assurance Department has developed an excel spreadsheet which processes and records the %TU and associated results. This spreadsheet automatically calculates %TU, determines data usability, and documents qualifier flagging for the sample batches containing matrix spike and duplicate data. Qualifier flagging for Laboratory Control sample (LCS) or Prep Blank quality control failure is manually recorded in the %TU excel spreadsheet. The usability of data in sample batches not containing matrix spike or duplicate quality control samples is determined manually on a batch by batch basis at this time. Likewise, compliance with detection limit controls and the percentage of qualified data in a batch is determined manually. Data completeness is determined from queried LIMS records and TU-usability determination summaries. Cogent evaluation of QC trend charts is awaiting finalization of the LIMS upgrade and customization.

A total of 432 analyte WorkSheets (sample batch data packets) were processed by the QA Department for this report. Reported data generated by non-Cotter laboratories is also recorded in the LIMS WorkSheets. A contract laboratory performed uranium in urine bioassay beginning in July 2010. This data has also been: subjected to Cotter

quality assurance review to the extent attainable by existing supplier-client agreements; is included in the total WorkSheet count; and has had the associated quality control data carried into the %TU spreadsheet.

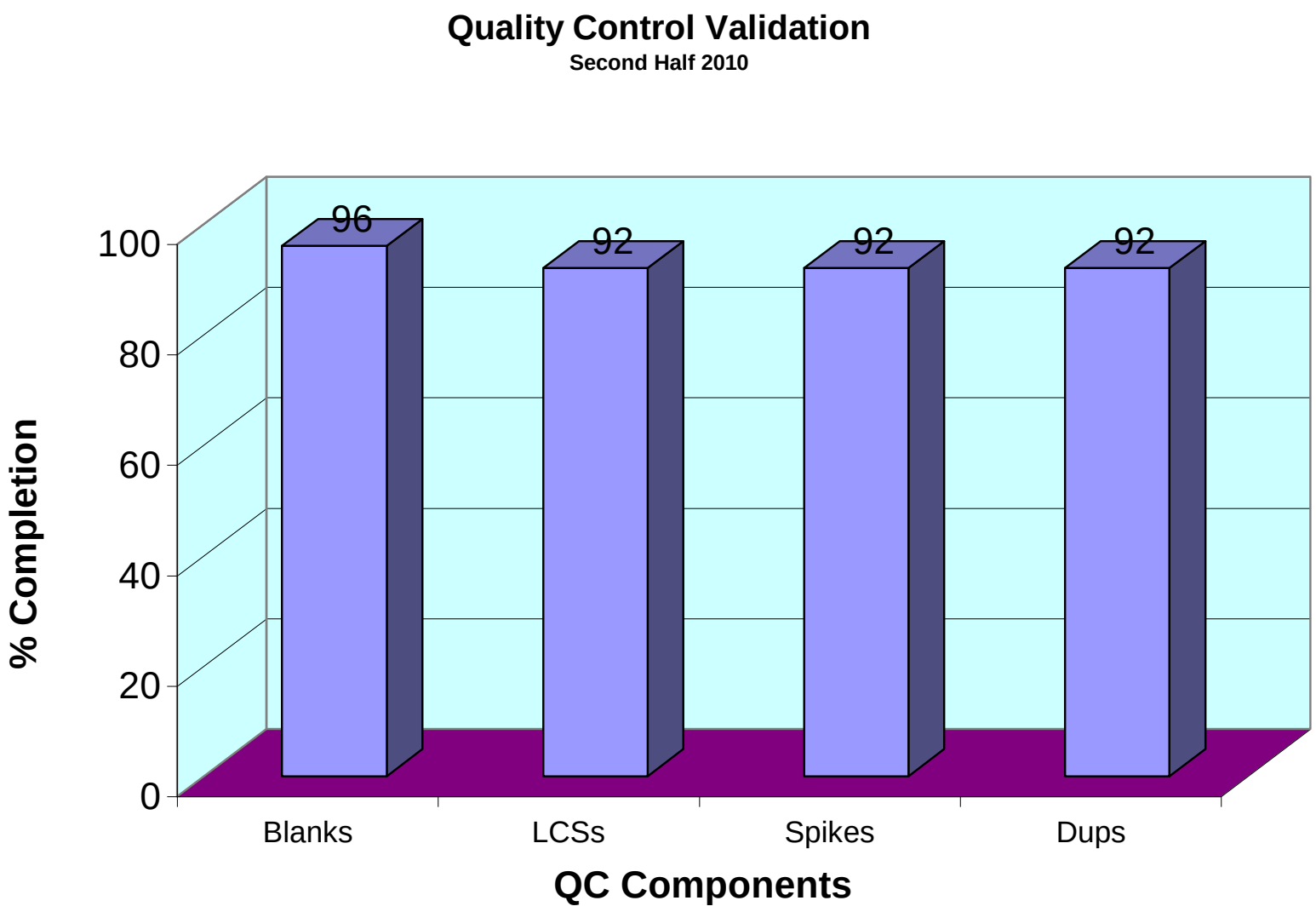
Quality review and assessment has been completed on most (see next paragraph) report data generated by the Cotter on-site analytical laboratory for samples collected during the third and fourth quarter monitoring periods of 2010. Sample batch data folders ("WorkSheet Data Packets") from 432 analytical sample batches were reviewed. 426 of these WorkSheet Data Packets were determined to contain usable data. 215 of them reported acceptable TU values. 211 did not allow TU calculation but were determined to be usable. Eleven WorkSheets contained flagged data. The data from five of the flagged WorkSheets was determined to be "usable". Data in the remaining six flagged WorkSheets was labeled "unusable". Five of these "unusable" sample batches consist of bioassay sample results reported by a contract laboratory. All five of the contract lab's sample batch results were identified as "unusable" because they failed to meet the routine Cotter QAPP Section 10.2.2 quality control acceptance criteria. The contract laboratory's QC acceptance criteria are broader. Three of the five batches were considered acceptable by the contracting lab's broader QC acceptance criteria. They were then identified as acceptable under the "alternate acceptance criteria" allowance of QAPP Section 10.2.2, but the "unusable" classification was retained in the TU spreadsheet. The sixth "unusable" WorkSheet contained results which were produced during development of a uranium isotopic ratio determination and were not used for report preparation. This sixth unusable WorkSheet also contained greater than 10% qualified data. QAPP Section 10.2.4 "Data Completeness" is 97.5%" for the 432 sample batches processed.

The Cotter Analytical Laboratory installed a Perkin Elmer NexION® 300X ICP-MS (Inductively Coupled Plasma-Mass Spectrometer) in early 2011. This ICP-MS will eventually assume the analysis of all metals and uranium previously conducted via AA (atomic absorption flame and graphite furnace analyses of metals) and KPA (Kinetic Phosphorimetric Analysis of uranium). A relatively small number of ICP-MS uranium and selenium results were produced by the Cotter Laboratory and are included in this report. There are four selenium in water, four uranium in water, and two uranium in environmental air sample batches analyzed by ICP-MS. At this time, these ten sample batches have not been released to the Quality Assurance Department for final quality review but have received internal laboratory review. It should be noted that although the written ICP-MS analytical procedure (Cotter Analytical Procedure SOP 2-200 Version: 01 3/2/11) has not yet received official approval by the State; the procedure was developed in consultation with Department personnel and has been submitted for approval.

The overall percentages of completion of the quality control validation at the time this performance report is being written are depicted on the accompanying graph PRQ1, *Quality Control Validation – Second Half 2010.* The depicted quality control validation completion percentages were conservatively estimated to be 96% for QC blanks and 92% for each of the other three QC data types. The reduced percentage completion

values can be attributed to three factors: (1) Final QA Department QA/QC review of the previously discussed ICP-MS sample results has not been conducted. (2) Generation and review of all trend charts had not been performed. and (3) Automated LIMS qualification of data is not yet implemented.

Graph PRQ1


Quality Control Validation
Second Half 2010
% Completion
100
80
60
40
20
0
96
92
92
92
Blanks
LCSs
Spikes
Dups
QC Components

QUALITY ASSURANCE VERIFICATION -

General quality assurance verification may include: evaluation of sampling and/or analytical procedures, program or project design and activities, and data processing. In addition to the review activities discussed previously, the data contained within the report undergoes other types of QA verification assessment. The basis for the additional data verification assessment may include historical knowledge of constituent levels, radiochemical equilibrium ratios, material-specific constituent proportions, known environmental conditions, or comparisons of final results to preliminary screening data. Data verification may also include evaluation of the reported detection limits versus required detection limits and the effect of any failed QC samples on data usability. Discovery of unacceptable, anomalous, or unexpected results may be followed by a review of field or lab records, investigation of sample acquisition, handling, preparation or analysis, or a request for reanalysis. The Cotter Quality Assurance Department provides oversight and documentation of required formal quality assurance follow-up.

A number of data quality investigations were documented by the Quality Assurance Department during the time period covered by this report. There were two Data Requests (DR) recorded. Both were for additional new analyses of third and fourth quarter occupational air samples or composites. Nine quality assurance related Data Verification Request - Assay Correction Form investigations (DVR/ACFs) were generated by data reviewers and data users. Seven of these DVR/ACFs dealt with environmental sample results for groundwater. One DVR investigated bioassay results reported by a contract laboratory. Another DVR investigated a Cotter metal result reported on a quality assurance proficiency test. All have been resolved except for the proficiency test result investigation. The DVR/ACFs addressed 33 sample results.

There were three quality assurance related Corrective Action - Improvement Requests (CAIRs) generated during this reporting period. One documented the inability of the LIMS to correctly determine the sample due date in some instances. The second addressed elimination of an out-dated procedural requirement to print or develop electronic analytical result reports. The third required investigation of the cause of the contracting laboratory's failure to achieve acceptable "% Error" results for bioassay control specimen samples. The QA Department also conducted and reported seven quality assurance assessment "Evaluation" reports. Five focused on environmental sampling and monitoring procedures and practices. Two focused on urinalyses (uranium bioassay) result evaluation. In addition, the Laboratory and Quality Assurance Departments investigated a glitch in the LIMS data processing and reporting of certain RDL values which require the application of dilution factors to MDL values in their calculation.

This Quality Assurance Program Report focused on evaluation of employee dose and environmental monitoring data quality. The related quality assurance verification assessed data that can be categorized roughly into the following three categories: Breathing Zone (BZ)/General Air (GA), Environmental Air, and Water Monitoring. The overall percentages of completion of quality assurance verification (BZ/GA 98%,

Environmental Air 90%, and Water Monitoring 95%) are depicted in graph PRQ2, *Quality Assurance Verification – Second Half 2010.* The BZ/GA verification is estimated at 98% rather than 100% to allow for possible overlooked data qualification that might have been identified and denoted by a more fully updated and implemented LIMS system. The percentage of completion of Environmental Air and Water Monitoring QA verifications are conservatively estimated to reflect the ten ICP-MS U and Se sample batches that did not receive Quality Assurance Department review and approval.

Data problems that were identified during the data quality assessments are summarized in the attached Error Analysis – Second Half 2010 Tables. Table PRQ1 addresses quality control validation issues. Table PRQ2 addresses formally documented quality assurance verification errors.

Graph PRQ2

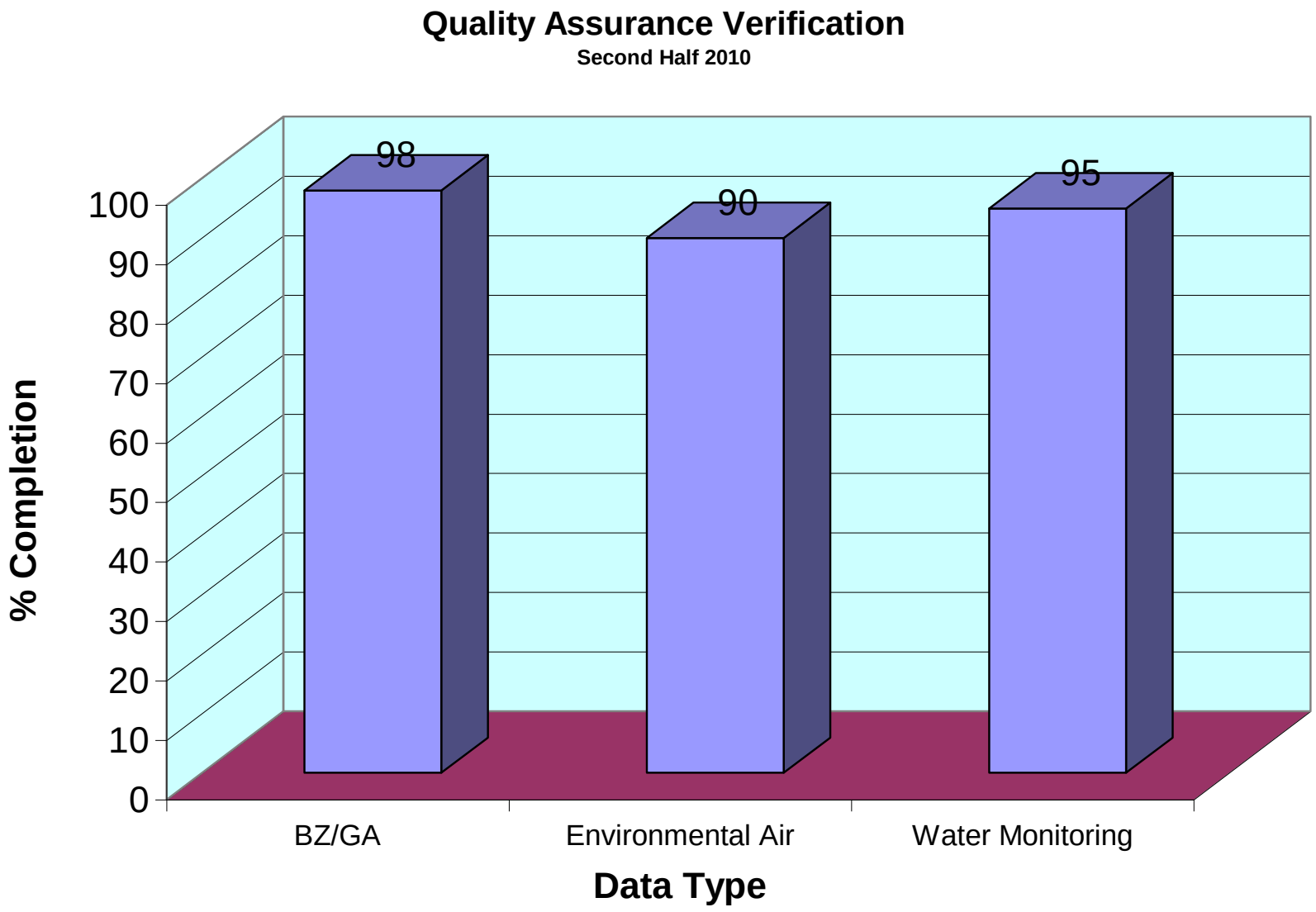

Quality Assurance Verification
Second Half 2010
98
90
95
100
90
80
70
60
50
40
30
20
10
0
% Completion
BZ/GA
Environmental Air
Water Monitoring
Data Type

Table PRQ1
Error Analysis - Second Half 2010

Quality Control Validation Issues

Description	Samples Affected	Frequency of Occurrence	Does Pattern Exist? Explain	Resolution
1) The methodology used to assess quality control accuracy and precision data in the LIMS (Aspen) is not consistent with the methodology prescribed in the QAPP. 2) Data qualification and usability determination performed manually only.	Blanks, Duplicates, Spikes, and LCSs.	Numerous	Yes. These discrepancies will continue to exist until the LIMS update/customization is complete and any required modifications to the QC processing sections of the LIMS have been completed.	Modification and customization of the Cotter LIMS is ongoing. The LIMS QC summary report is currently only used to verify proper data entry/data upload into LIMS. Until the upgraded LIMS is modified/customized and proven to meet all requirements for full QC assessment; QC review and approval will continue to be performed using calculations documented on the lab analysis result (LAR) printout in the data packet. Qualifier assignment, TU calculation, data usability determination, and data completeness calculation are performed by the QA Department at this time.
Manual data transfer and data input can result in erroneous information in analytical documentation and/or in the LIMS. (DVR 11-15-2010-2)	All data	< 5%	No. Due to the large volume of data that is manually transferred/input occasional random data entry errors are likely.	These types of errors are detected during internal laboratory and QA/QC data reviews. Corrections are made to the documentation and/or the LIMS database, as necessary. Electronic data transfer should greatly reduce these types of errors. The on-going modifications to the LIMS include implementation of electronic data transfer wherever possible.
Incorrect RDL value reported by LIMS. (Noted during QA Department quality review of WorkSheet No. 10-10640 Data Packet.)	Some analytes requiring dilution factor processing for RDL calculation.	24 Sample batches	This error may occur in some analyte reports where RDL is processed from MDL and a dilution factor.	The error was discovered during detection limit evaluation of WorkSheet Data Packet. In past LIMS input was modified to force correct RDL. A data processing correction will be required as an item in the LIMS update.

Table PRQ2
Error Analysis - Second Half 2010

Documented Quality Assurance Verification Errors

Document or Record	Type	# Results Corrected	Apparent Error Type	Results of Investigation	Does Pattern Exist? Explain
DVR/ACFs: 11-01-2010-1, 11-30-2010-1, and 2-09-2011-1 and 9-17-2010-1.	Groundwater and Bioassay	8	Anomalous results requiring reanalysis.	Reanalysis was required followed by correction in laboratory WorkSheet Data Packet and LIMS database.	This problem is usually detected during data user QA review of results and is usually followed by a request for reanalysis. Only formal documented requests resulting in sample result changes are listed here.
DVR/ACF 11-15-2010-2	Groundwater Field Data	3	Data identification and input error - Depth-to-water field data was mis-identified when transferring from field notes to SSTF and prior to input into LIMS.	Field data records were voluminous and complex. Incorrect interpretation of field data in field notes.	No. This is an unusual problem.

Note: An additional 20 analytical results were investigated and verified through the DVR/ACF documentation and required no change in reported values.

PERFORMANCE REPORT

Laboratory Program - July - December 2010

This report describes the types and numbers of analytical determinations as well as the dates the results were posted to the Laboratory Information Management System (LIMS). All of the data was available by March 31, 2011. Some of the measurements, for example, “perimeter” or environmental air sampler filter paper weights and urinalysis for uranium, are typically posted within a week or two of collection.

Gross alpha/beta breathing zone and general area air samples filters were collected and submitted by Radiation Safety Department technicians for assessment of airborne concentrations relative to the ALARA program and to monitor occupational dose. The sample load resulting from this analysis was one thousand seven hundred fifty (1750) samples for the second half of the year. Preliminary results were either available within twenty-four (24) hours of sample receipt or by the next business day for samples collected on weekends. Final analysis of these samples in all cases was completed well within the report preparation requirements.

Analytical Work Summary -

- Third (3rd) and fourth (4th) quarter anion analyses - one thousand five hundred fifty six (1556) analyses entered into LIMS no later than 01-07-11
- Three hundred forty five (345) perimeter (environmental) air sampler filter weights each weighed twice (690 data points) entered into LIMS by 01-07-11.
- Occupational air, perimeter air, and water for total uranium - 274 analyses by KPA plus 33 analyses by ICP-MS totaling three hundred seven (307) sample analyses entered into LIMS by 3/31/11.
- Perimeter air and water analyses for ^{226}Ra - seventy nine (79) sample analyses.
- Perimeter air and water analyses for ^{210}Pb - sixty nine (69) sample analyses.
- Water analysis for ^{210}Po - thirty-nine (39) sample analyses.
- Occupational air, perimeter air and water analyses for ^{230Th} - one hundred eighty-two (182) sample analyses.
- Occupational and perimeter air analyses for ^{232}Th - one hundred thirty one (131) sample analyses.
- Uranium in urine analyses - eight hundred thirty (830) sample analyses including six hundred eighty-eight (688) analyses performed by Energy Laboratories Inc.
- pH and conductivity of water sample determinations - one hundred twenty six (126) sample determinations.
- Gross alpha & beta analyses of sediment - three (3) samples analyses entered into LIMS by 3-31-11.
- Third (3rd) quarter analyses for metals: Ca, Fe, K, Mg, Mn, Mo, Na, and Se - eight hundred thirty nine (839) analyses by Cotter and 53 Se analyzed by ALS Laboratory Group entered into LIMS by 11-29-10

- Fourth (4th) quarter analyses for metals: Ca, Fe, K, Mg, Mn, Mo, Na and Se - eight hundred seventy six (876) analyses by atomic absorption (AA) and 60 Se analyses by ICP-MS entered into LIMS by 03-31-11.
- Water samples sent to ALS Laboratory Group for Se analysis - 53 sample analyses.

The Cotter Laboratory encountered significant problems with instrumentation and subsequently lost analysis time during the report period. The Kinetic Phosphorimetric Analyzer's laser failed and the KPA became unusable in late December 2010. This resulted in a backlog of uranium analysis. The Cotter Analytical Laboratory acquired a Perkin Elmer NexION® 300X ICP-MS (Inductively Coupled Plasma-Mass Spectrometer). This instrument was supposed to assume all uranium and metal analyses that the KPA and Atomic Absorption instruments had been performing. To meet the needs of the ICP-MS, extensive modifications were made to portions of the laboratory building. Instrument installation began February 1, 2011. After installation and initial start-up the new ICP-MS unit failed. On February 16th the first Cotter Nexion 300X ICP/MS was declared not repairable by Perkin Elmer and a replacement was ordered. The replacement was shipped to Cotter and installed during the week of March 7th. The second ICP-MS was started up and lab technician training was conducted the week of March 14th. The replacement ICP-MS was deemed operational and put into preliminary analytical service the week of March 21st. This semiannual report contains some uranium and selenium analyses produced by Cotter's new ICP-MS instrumentation.