QUALITY ASSURANCE/QUALITY CONTROL

OVERVIEW OF QUALITY ASSURANCE PROGRAM PLAN (QAPP)

The quality assurance and quality control activities for the environmental and occupational sampling and monitoring studies at the Cotter Corporation, Canon City Mill Facility (CCMF) are described in the Quality Assurance Program Plan (QAPP). Throughout 2013 quality assurance program guidance was provided by the May 22, 2009 Revision of the QAPP officially titled: 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. This current version of the QAPP incorporates U. S. Environmental Protection Agency (EPA) quality plan guidance and Data Quality Objectives. The QAPP is applied to activities mandated by license conditions, environmental and occupational monitoring, and remediation related projects including those required by the Remedial Action Plan (RAP) of 1988.

The Program Performance Criteria recorded in the QAPP establish the expectations of quality control data processing. Specific to the analytical laboratory, the QAPP’s Program Performance Criteria require monitoring of: “Total Uncertainty” of accuracy and precision sample results; “Usability” of sample batch results, percent of “Qualified” or “Flagged” data, “Completeness” of data, and analyte detection limits. In addition to laboratory quality assurance, the Program Performance Criteria reinforce requirements for performance evaluation, corrective action and reporting of monitored activities.

MONITORING PROGRAM AND GENERAL QUALITY ASSURANCE

The Radiation Protection Plan Procedures (RPPP) prescribe appreciable QA Department involvement in the Environmental/Radiation Department’s activities. For example the Cotter Analytical Laboratory normally performs routine uranium in urine analysis (“urinalysis”) as support to the CCMF bioassay program. That program is described in RPPP RH-050 and is intended to meet specifications of the Nuclear Regulatory Commission (NRC) Reg. Guide 8.22. The QA Department manages the “Control Urine Specimen” portion of the bioassay program established by Reg. Guide 8.22, Section 8. This QA function has included preparing and submitting control specimen samples then monitoring control specimen data for indications of biases and trends in the urinalysis results. Occupational and Environmental Monitoring Procedures are routinely the subject of QA Department audits and are formally documented in QA Evaluation Reports. Any subsequent required corrective actions or need for procedural improvements become the subject of formal requests documented in the QA Department’s Corrective Action - Improvement Request System.

Program Assessments (Evaluations)

The Quality Assurance Department staff is charged with auditing laboratory analysis procedures, performance evaluation results, and written monitoring program (RPPP) procedures. This process is referred to as “Evaluation” in Section 11 of the QAPP. The evaluation process assesses the correctness of the performance of the procedure by the person conducting the work
and can also evaluate the effectiveness and adequacy of the procedure in general. Incorporation of the Corrective Action – Improvement Request (CAIR) system and the QA Department’s program assessment function provides a process to identify and correct deficiencies or affect improvement to the item being audited.

A total of nineteen (19) formal evaluation reports were prepared either during 2013 (thirteen) or subsequently on 2013 work product (six). The program assessment “evaluation” process led to issuance of one (1) of the six (6) formally documented Corrective Action - Improvement Requests recorded in 2013.

**Training and Qualification Documentation**

Radiation Protection Program Procedure training, QA Program Procedure training, and specially approved training and/or certification are normally documented through written notification to the Vice President, Mill Operations and/or the Quality Assurance Coordinator (“QA Officer”) using the QA Department’s *Personnel Qualification Form* (Form QAPP-1). The QA Coordinator maintains a copy of each of these documents in his Personnel Qualifications file.

The Laboratory Manager maintains a Laboratory Personnel Training And Qualifications Documentation System for his analysts and technicians. He determines the requirements for satisfactory performance of each analytical procedure or task. He maintains personnel qualifications via a checklist that is specific for individual job tasks. Once an analyst has met all basic requirements and has demonstrated satisfactory performance on the specific job’s tasks, the Laboratory Manager completes the checklist and places it in that individual’s laboratory training qualification file. The checklists are updated yearly or as needed. The Lab Manager maintains the Laboratory Personnel Training and Qualification files in his office in the Analytical Laboratory.

The Radiation Safety Clerk maintains the Training Records Documentation and Tracking (TRDT) Program on the shared drive located at Z:\Environmental_Radiation\Working Folders\CCMF\My Documents 2013\Training. The training subfolder contains an Excel workbook, “*Cotter Employee Training Worksheet 2013*”, and other media which document and track the required personnel training in the areas of Radiation Protection, Health and Safety, Quality Assurance, Radiation Work Permit, MSHA training or certification, respirator certification, the “Read Only” Training Program, and any additional special training or certification. The Read-Only Training program documents only that the individual has read the procedure, revision, or other printed material. Read-Only Training is applied to all employees for documenting required pertinent procedural training and is often used as the initial step in the formal training process. The CCMF training records documentation contains completed records through 2013. Formal training session records are filed in the “RADIATION SAFETY - Radiation Safety Training Data” file. The Certificates of Training are filed in individual personnel files when provided.

**Equipment and Instrument Calibration**

Laboratory equipment is calibrated daily or at required frequencies. Radiometric counting calibrations are stored electronically and printed as required. Counting equipment calibration documentation is maintained in the lab and is available for review either in electronic format or hard copy. The non-radiometric Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) is
calibrated daily. Calibration records are printed for each ICP-MS sample batch (identified in the LIMS by the moniker “WorkSheet”) and are included in each sample batch’s “WorkSheet Data Packet.”

The Assistant Radiation Safety Officer (ARSO) inventories the availability of operable and calibrated survey and monitoring equipment outside the laboratory. The ARSO documents and tracks the equipment/instrument calibration status on his assigned computer in the Excel workbook, “Instrument Calibration-Inventory.xlsx”. Data for each survey or monitoring device’s record is entered into this workbook. Records include the equipment description, maintenance and calibration record, and calibration schedule. The Radiation Safety Department personnel determine which instruments are due for calibration and initiate follow-up. Hardcopies of calibration, maintenance, and related records for all radiation monitoring and survey equipment are located in the “Radiation Safety 2013 Calibration”, “Radiation Safety 2013 Calibration – Outside Vendor”, and the “Radiation Safety 2013 Calibration - Survey Instruments” folders in the 2013 Radiation Safety file cabinet drawers in the Radiation Safety – RAP Records Storage Room of the Office/Laboratory Building. The Survey Instrument Calibration Forms are received from the Radiation Safety Technicians when completed and are filed in the “Radiation Safety 2013 Calibration – Survey Instruments” folder.

Source Inventory

Sources used in the laboratory for radiochemical analyses and counting instrument calibration are stored within the laboratory. They are identified and documented in the “Radiochemistry Solutions Logbook (solutions) and on either the “Solution and Solid Sources Inventory” or “Source Inventory” sheets. Both source inventory sheets are stored electronically within the lab’s Counting Room computer and backed up on the lab’s V drive. The lab’s sources are inventoried monthly during the QA Department’s Monthly Laboratory Inspection. The “Check Source Inventory Report” lists check sources used to assure proper operation of each radiation detection instrument. This inventory is maintained by the Radiation Safety Department. The Check Source Inventory Report can be found in the “Sources Information” binder on the Radiation Safety Department’s book shelves in the Radiation Safety – RAP Records Storage Room of the Office/Laboratory Building. An electronic version of the Check Source Inventory Report can be found on the shared drive - Z:\Environmental_Radiation\Check Sources\Check Source Inventory Report Updated 4-3-2014.xls.

ANALYTICAL LABORATORY QUALITY ASSURANCE AND QUALITY CONTROL

Major Analytical Laboratory Instrumentation

Throughout 2013 uranium and metal analyses were conducted using the Perkin Elmer NexION® 300X ICP-MS (Inductively Coupled Plasma-Mass Spectrometer) unit. Radiochemical analysis data was produced by either the ORTEC Octete Plus Counting System in conjunction with TENNELEC Alpha spectrometers or the Protean Low-Background Gas Proportional Counters and appropriate software.

Analytical Procedures (SOPs) and Quality Assurance

The Cotter Analytical Laboratory developed or revised and then submitted eight (8) Analytical
Laboratory Procedures, SOPs 1-3, 1-4, 2-201, 4-2, 4-3, 4-4, 4-9, and 5-10 during 2013. Cotter is awaiting final approval of these procedures from the licensing agency. The individual written analytical laboratory procedures describe QA and QC details specific to the corresponding analytical procedure or determination. When applicable, the quality details cited in the laboratory procedures take precedence over the broader or more generalized QA/QC guidance stated within the QAPP. The analytical laboratory procedures are reviewed for update revisions each two years.

Data Management

Explicit data such as sample field measurements and analytical laboratory results are recorded and stored in the Laboratory Information Management System or “LIMS”. The LIMS is a computer database application system dedicated to the management of sample information. Update and customization of the LIMS and electronic data transfer had been the focus of much discussion and investigation since the latest version of the QAPP was adopted. The Program Performance Criteria of the QAPP set data processing expectations. Many of these involve complicated statistical evaluation, data testing, data qualification tracking, and data reporting requirements. Comprehensive QC sample evaluation is complex and beyond the capability of the current LIMS algorithms. Many of the data evaluations involve complex judgment decisions by involved personnel at various stages of the sample and data processing and require knowledgeable personnel oversight. The data management expectations are being met through the functionality of the current LIMS and manual hands-on data processing and documentation by QA Department staff. Previous plans for totally automated LIMS data management have been abandoned in favor of continued manual data review, qualification, and validation by QA Department personnel utilizing improvements to existing data processing practices and customized tools developed for our specific application. Improvement in utilization of electronic data transfer is an on-going effort.

DATA QUALITY INDICATORS

Data Verification and Corrective Action

There is the potential for occasional anomalous results to be detected in all laboratories’ reports. In such instances the Data Verification Request/Assay Correction Form (DVR/ACF) record system is implemented to document the specific concerns, required investigation, and any follow-up on questioned results.

The Quality Assurance Program also employs a Corrective Action and Improvement Request (CAIR) documentation system to formally document requested corrective action or system improvement when a need is identified. This system also documents the follow-up taken in response to the initial request. The QA Department’s CAIR system has been applied to monitoring and support activities beyond data production.
Laboratory Performance Evaluation

The CCMF’s Quality Assurance Department conducts quarterly Inter-laboratory Exchange and semiannual Intra-laboratory Data Comparison Programs. These programs require the Environmental Department to collect a sample of groundwater from which replicate splits are submitted to laboratories participating in the performance evaluation program. In both programs the laboratories’ results are compared statistically and evaluated. Inter-Laboratory Exchange (ILX) replicate samples are submitted to the Cotter Analytical Laboratory and three (3) participating commercial laboratories. The Inter-laboratory Exchange Program is conducted primarily to demonstrate the accuracy of the Cotter Analytical Laboratory’s results in comparison to those of participating commercial laboratories performing similar analyses. In doing so systematic errors in the laboratories’ results can be detected. The 2013 ILX studies noted an apparent continued variability of iron result; especially those reported by one of the participating non-Cotter laboratories and one-time occurrences of high biases in Cotter sulfate and selenium results. In the Intra-laboratory Comparison (ILC) program, a number of replicate samples are submitted to the Cotter laboratory for precision and variability evaluation. In the final analysis of 2013 ILC results, Th-230, TSS, and Se could be worthy of continued laboratory monitoring. However their very low reported values and their proximity to their respective LLDs diminish the significance of precision assessment for these analytes.

The Cotter laboratory also participates in two (2) types of proficiency test (PT) studies - “Uranium and Radium in Water” and “Water Pollution”. These PT studies are supplied by an accredited proficiency test provider. Any of the Cotter lab’s Uranium and Radium in Water and/or Water Pollution proficiency test results that do not initially receive an “Acceptable” performance evaluation by the PT provider become the subject of a formal data verification investigation (DVR). The Cotter laboratory participated in two “Uranium and Radium in Water” and three “Water Pollution” PT studies in 2013. Four water pollution proficiency test results submitted by the Cotter Analytical Laboratory in 2013 received “Not Acceptable” performance evaluations and triggered a DVR/ACF investigation, follow-up root cause analysis, and formal corrective action.

The Cotter Analytical Laboratory also demonstrates performance acceptability and the quality of reported analyte values through participation in the annual EPA NPDES Discharge Monitoring Report–Quality Assurance (DMR-QA) Study and through its CLIA (Clinical Laboratory Improvement Amendments) Certification.

Detection Limits

License Conditions 25.2.2 and 27.2 reference the lower limits of detection (LLDs) that are to be followed for monitoring sample analyses. The non-radiometric detection limit determination methodologies for Minimum/Method Detection Limits or “MDLs” are conducted at least annually and their results are reported to the Laboratory Manager when the determinations are performed. The MDLs of the non-rad analytes including the target elements molybdenum and uranium are normally determined early in the year and referenced during subsequent sample batch analytical result quality review/evaluation. The Quality Assurance Department conducts a quality review and verifies compliance with the non-radchem target element MDLs during the data validation process. The radiochemical analytical procedures or “SOPs” describe the relevant detection limit computations. The software processing the radiometric result data performs the
detection limit calculations for each reported result. A “Minimum Detectable Activity or MDA” is reported for each radiochemical analysis. The radiochemical MDAs, often referred to as “LLDs”, calculated for each sample analysis are evaluated by the Laboratory Manager as he performs his quality review and approval of the sample batch analytical data packages. The MDLs and MDAs/LLDs are recorded somewhere within each sample batch’s “WorkSheet Data Packet” - either within the packet’s raw data or on the analyst’s worksheet. Any MDL/LLD determination values found to not meet license condition requirements require follow-up investigation or corrective action.

**QA-QC Review**

As a part of the quality oversight of sampling, the QA Department reviews sample chain of custody and associated Laboratory Information Management System (LIMS) log-in documentation of Cotter samples generated for environmental or occupational monitoring reporting. The “Cotter Analytical Laboratory QA-QC Data Review Guidelines” summarize the quality control review requirements of all Cotter laboratory analyses types. These guidelines are used to assure that critical data components are assessed during review of each sample batch data packet. Standard laboratory analysis quality control and quality assurance operations are built into the analytical routines employed by the CCMF Laboratory. All monitoring sample batches contain the appropriate blank, spike, control standard and duplicate samples required to provide sample accuracy and precision control guidance.

Summaries of the quality control verification and quality assurance validation activities are prepared on a semiannual basis and reported in the 2013 Semiannual Reports: *Performance Report 1st half 2013 8-29-13*” and “Performance Report 2nd half 2013 2-28-14” submitted to the CDPHE. These summaries address the QC validation and QA verification of analytical data processed by Cotter’s analytical laboratory during the reporting period. Relevant data verification and corrective action requests are summarized in these reports. An error analysis is also included in each report. A portion of each performance report provides a graphical representation of the percent of completion at the time of report preparation of the quality analysis of specific data types discussed in other portions of the semiannual report package.

**QUALITY ASSURANCE PROGRAM MANAGEMENT AND RESPONSIBILITIES**

The 2009 revision of the QAPP redefined and rearranged quality assurance management responsibilities. The designation of a “Quality Assurance Manager” was eliminated and the former Quality Assurance Coordinator was re-designated as “Quality Assurance Officer”. The Vice President, Mill Operations assumed many of the responsibilities of the Quality Assurance Manager that were described in previous versions of the QAPP. The Quality Assurance Officer inherited all of the previous Quality Assurance Coordinator’s duties and responsibilities and the QA Manager’s administrative duties that were not reassigned to the Vice President, Mill Operations. Despite these revisions, formal meetings called by the Quality Assurance Officer (“QA Coordinator”) to address quality issues at the CCMF are still referred to as “QA Manager’s Meetings”. Topics addressed at QA Manager’s Meetings held in 2013 included: use of data qualifiers, follow-up and corrective action for “Not Acceptable” proficiency test evaluations, and auditing of spreadsheets used in report preparation. A series of informal QA meetings were also conducted in 2013 to evaluate and plan the implementation of quality assurance and quality
control activities relevant to the environmental radon monitoring program. These informal meetings were not identified as QA Managers Meetings.