Colorado Oil and Gas Conservation Commission

Sampling and Analysis Plan for Naturally Occurring Radioactive Material in Oil and Gas Well Drill Cuttings

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1.0 Introduction, Purpose, and Approach

Colorado Oil and Gas Conservation Commission (COGCC) seeks to understand the concentration of naturally occurring radioactive material (NORM) in solid and liquid exploration & production (E&P) related wastes generated during drilling and production activities. This Sampling and Analysis Plan (SAP)* has been developed to provide the COGCC staff and contractors procedures for the collection of representative E&P materials for the determination of NORM.

Throughout Colorado many companies perform oil and gas (O&G) activities, drilling through various subsurface layers of rock to produce oil and natural gas. Drill cuttings and produced water are potentially impacted by NORM and may include elevated concentrations of radium-226 (Ra-226), a common NORM radionuclide associated with naturally occurring uranium. For purposes of this plan any water emerging from the well is considered to be produced water even if was previously injected for hydraulic fracturing or other purposes. An emphasis will be made to focus produced water sampling on formation waters; in some cases evaluation of stimulation fluids will be included.

Sampling activities, in order of priority, will include:

- Drill cuttings;
- Produced water.

This SAP defines protocols for radiological surveys, sampling, chain-of-custody (COC) procedures, offsite radiological laboratory analyses, and quality assurance practices.

*In April 2013 Pennsylvania Department of Environmental Protection contracted with Perma-Fix Environmental Services, Inc. (PESI) to perform a comprehensive a study of NORM and TENORM related to the oil and gas exploration activities throughout the Commonwealth of Pennsylvania. As part of this effort PESI prepared a sampling and analysis plan. (PESI 2013a, 2013b). This SAP borrows extensively from those documents.

2.0 Site Description

2.1 Background

The sampling locations associated with oil and gas drilling and production will vary from site to site throughout the State of Colorado. This initial effort will be concentrated in the Greater Wattenberg Area of Northeastern Colorado and the Raton Basin of Southeastern Colorado.

2.2 Site Characterization

Primary Constituents of Concern (COCs) for oil and gas production radiological characterization include uranium (U-238, U-235 and U-234), thorium-232, radium (Ra-226 and Ra-228), radon (Rn-220 and Rn-222) and any unsupported decay chain radionuclides.

3.0 Scope and Objectives

This study is to investigate the possible occurrence of NORM in E&P related waste materials from O&G activities. Information on waste volumes and levels of radioactivity will be informative to the management of these materials. Radon in produced natural gas is not in the scope of this study.

Radiological support surveys and sampling performed for this NORM study will consist of:

- Sampling of various media for off-site radiological and laboratory analyses.

This section summarizes the scope and objectives of these survey activities; detailed descriptions of field screening and sampling procedures are presented in Sections 5.0 and 6.0, respectively.

3.1 Scope of Field Screening Activities

Field screening activities will consist of using field instruments, such as exposure rate meters and surface contamination meters to identify the possible presence and degree of NORM in cuttings and produced water. Screening of both solid and aqueous samples will be performed.

Direct-read radiological instruments and detectors will be used throughout the field operations of the project for scanning and surveying of personnel, equipment, materials and areas. The instruments and detectors will be operated and maintained by the COGCC staff or contractors. Instruments, detectors, and equipment (or their equivalents) that may be used on-site during field screening are listed in Table 1.

Table 1. Field Screening Equipment

Instrument	Detector	Use
Ludlum Model 2, or equivalent	Ludlum Model 44-9 Pancake G-M Probe	General purpose survey meter for surface contamination measurement
Ludlum Model 192 Micro-R Meter, or equivalent	2” x l” Nal Scintillator	Exposure rate survey meter for high energy gamma rays

3.2 Scope of Field Sampling Activities

Field sampling will consist of collecting samples of environmental media from sources for off-site laboratory analysis. The following types of field samples will be collected for the purposes identified:

- Drill cuttings for off-site radiological laboratory analyses for NORM characterization.
- Flowback and produced water samples for off-site radiological laboratory analysis for characterization purposes.

Off-site analyses of solid and aqueous samples may include the following parameters as specified in Section 6.0:

- Gross alpha and beta
- Gamma spectroscopy to identify radionuclides
- Alpha spectroscopy for uranium (U-238, U-235, and U-234), thorium (Th-232, Th-230 and Th-228), and for any unsupported decay chain radionuclides

- Alpha scintillation for radon (Rn-220 + Rn-222)
- Radium (Ra-226) by Gamma spectroscopy after ingrowth (solids)
- Ra-226 by GFP or emanation method (aqueous)
- Alpha spectroscopy of Pb-210 and Po-210
- Beta analysis for Ra-228
- Metals analysis of Uranium and Thorium

Minimum Detectable Activity (MDA) for laboratory analysis is defined in Table 2. Sampling and laboratory analysis requirements are summarized in Table 3. Sample characteristics including sample size, containers, preservation and holding times are summarized in Table 4.

4.0 Field Sampling Activities

Specific sampling parameters, laboratory analytical methods, and numbers of samples are discussed further in Section 6.

4.1 Solid Sample Type and Locations

The solid samples to be collected consists of drill cuttings as produced on a drill rig including cuttings stored temporarily or land applied cuttings. Drill cuttings (solid material) from both the vertical and horizontal drilling phases will be sampled by geologic unit or portion thereof and may also be collected from land application sites.

4.1.1 Solid Sampling Methods

Solid samples will be collected using clean (decontaminated) reusable or new disposable sampling tools. Sampling tools may be decontaminated prior to first use on-site, between sampling locations, and following last use on-site (i.e., before demobilizing that equipment) as appropriate. The samples selected for analysis will be placed into laboratory approved containers immediately following collection and labels promptly affixed to the sample containers. The samples will be transported via delivery service or by the sampling crew under chain-of-custody control to the off-site subcontract laboratory for analysis. Table 4 identifies container types that will be used for collection of these samples. Minimum sample quantities required for laboratory analysis are also identified in Table 4.

If materials are present in multiple contiguous intervals, a compost of the materials will be homogenized in a stainless steel bowl or disposable aluminum pan to the best that the material will allow. A sample will be collected from the homogenized material and sent for the appropriate analysis. Homogenization of individual interval samples by the laboratory may be utilized instead of field homogeniztion

Additional sample preparation as specified by the laboratory for specific analyses may be required. For gamma spectroscopy usually no field prep is necessary. However, for gamma spectroscopy, alpha spectroscopy and gross alpha and beta analyses additional sample preparation may be necessary in the laboratory.

4.1.2 Laboratory Analysis of Solid Samples

Solid samples will be analyzed by gamma spectroscopy to identify NORM radionuclides following size reduction, homogenization and ingrowth period.

4.2 Aqueous Sample Type and Location

Grab samples may be opportunistically collected on drilling sites from locations outlined below:

- Flowback and produced water;
- Temporary water storage vessels (such as produced water tanks).

4.2.1 Sampling Methods for Aqueous Samples

A representative grab sample will be collected from the appropriate tank/outlet directly into the sample containers using available piping and valves. If necessary, a disposable bailer may also be used to collect the sample and the contents of the bailer added directly to sample containers. Samples will be placed into laboratory-prepared containers immediately following collection and caps and labels promptly affixed to the sample containers. In cases where sample valves are available, samples may be collected directly into approved sample containers. The samples will be transported via overnight delivery service under chain-of-custody control to the off-site subcontract laboratory for analysis or transported to the on-site lab. Table 4 identifies container types to be used for collection of these samples. Upon receipt, the lab will check pH of containers with HNO_3 preservation and will acidify if pH>2 is measured.

4.2.2 Laboratory Analysis for Aqueous Samples

The samples will be analyzed for gross alpha and beta, alpha spectroscopy, liquid scintillation and by gamma spectroscopy to identify radionuclides. Laboratory analysis methods for these parameters are identified in Table 3.

4.3 Sampling Equipment Decontamination Procedures

Disposable sampling equipment will be used wherever possible to minimize decontamination requirements. When reusable equipment is used, such equipment will be decontaminated both prior to sampling in the field and between uses, as appropriate. The following decontamination steps will be performed for reusable equipment, in the following order as necessary:

- Potable water rinse;
- Wash with laboratory-grade detergent (Alconox ®, Liquinox® or equivalent);
- Distilled water rinse; and
- Air drying.

5.0 Documentation

5.1 Field Documentation

Information pertinent to field activities will be recorded on field logbooks. The logbooks will be bound and the pages will be consecutively numbered. Sufficient information will be recorded in the logbooks to permit reconstruction of site sampling activities. Information recorded on official project documents (e.g., survey forms, chains-of-custody, etc.) will not be repeated in the log books except in summary form or cross-reference notation where determined necessary. Field log books will be kept in the possession of the appropriate field personnel, or in a secure place when not being utilized during field work. Entries recorded in log books will be made in blue or black, waterproof ink and may include, but not be limited to, the following information:

- Surveyor/sampler, date, and times of arrival at and departure from the site;
- Description of the field activity and summary of daily tasks;
- Names and responsibilities of field crew members;
- Sample collection method and number/volume of sample(s) collected;
- Information regarding activity changes and scheduling modifications;
- Field observations and weather conditions;

- Types of field instruments used and purpose of use, including calibration methods and results;
- Field measurements made and quantities/volumes of material sampled;
- Scanning/surveying of equipment and materials;
- Global Positioning System (GPS) coordinates, as appropriate.

Additionally, the sampler will record any other pertinent data during each sample event, e.g., influent volume from shale, total influent flow, and effluent flow.

Field data sheets and Radiological Survey forms may be used to record field information in addition to the use of log books.

5.2 Sample Documentation

5.2.1 Sample Numbering System

A unique sample numbering scheme will be used to identify each sample collected and designated for on-site and off-site laboratory analysis. The purpose of this numbering scheme is to provide a tracking system for the retrieval of analytical and field data on each sample. Sample identification numbers will be recorded on sample labels or tags, field data sheets and/or logbooks, chain-of-custody records and all other applicable documentation used during the project.

5.2.2 Sample Labels

Labels will be affixed to all sample containers during sampling activities. Information will be recorded on each sample container label at the time of sample collection. The information to be recorded on the labels will be as follows:

- Sample identification number;
- Sample type (discrete or composite);
- Site name and area/location number;
- Analysis to be performed;
- Type of chemical preservative present in container;
- Date and time of sample collection; and
- Sample collector's name and initials.

5.2.3 Cooler Receipt Checklist

The condition of shipping coolers and enclosed sample containers will be documented upon receipt at the analytical laboratory. This documentation will be accomplished using a cooler receipt checklist utilized by the contract laboratory.

5.2.4 Chain-of-Custody Records

Chain-of-custody procedures implemented for the project will provide documentation of the handling of each sample from the time of collection until completion of laboratory analysis. The chain-of-custody form serves as a legal record of possession of the sample.

Custody will be documented throughout the project field sampling activities by a chain-of-custody form initiated each day during which samples are collected. The chain-of-custody form will accompany the samples from the site to the laboratory and will be returned to the laboratory coordinator with the final analytical report. Personnel with sample custody responsibilities will be required to sign, date and note the time on a chain-of-custody form when relinquishing samples from their immediate custody (except in the case where samples are placed into designated secure areas for temporary storage prior to shipment). Bills of lading or air bills will be used as custody documentation during times when the samples are being shipped from the site to the laboratory, and will be retained as part of the permanent sample custody documentation.

Chain-of-custody forms will be used to document the integrity of all samples collected. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, chain-of-custody forms will be filled out for sample sets as determined appropriate during the course of fieldwork.

The individual responsible for shipping the samples from the field to the laboratory will be responsible for completing the chain-of-custody form and noting the date and time of shipment. This individual will also inspect the form for completeness and accuracy. After the form has

been inspected and determined to be satisfactorily completed, the responsible individual will sign, date, and note the time of transfer on the form. The chain-of-custody form will be placed in a sealable plastic bag and placed inside the cooler used for sample transport after the field copy of the form has been detached. The field copy of the chain-of-custody form will be appropriately filed with the project files.

Chain-of-custody seals will also be placed on each cooler used for sample transport by commercial courier. These seals will consist of a tamper-proof adhesive material placed across the lid and body of the coolers. chain-of-custody seals will be used to ensure that no sample tampering occurs between the time the samples are placed into the coolers and the time the coolers are opened for analysis at the laboratory. Cooler custody seals will be signed and dated by the individual responsible for completing the chain-of-custody form contained within the cooler.

5.2.5 Receipt of Sample Forms

The subcontract laboratory will document the receipt of samples by accepting custody of the samples from the approved shipping company. In addition, the subcontract laboratory will document the condition of the environmental samples upon receipt.

5.2.6 Documentation Procedures

The tracking procedure to be used for documentation of all samples collected during the project will involve the following steps:

- Collect and place samples into laboratory sample containers;
- Complete sample container label information;
- Complete sample documentation information in the field logbook;
- Complete project and sampling information sections of the chain-of-custody form(s);
- Complete the air bill for the cooler to be shipped to off-site laboratory, if applicable;
- Perform a completeness and accuracy check of the chain-of-custody form(s);
- Complete sample relinquishment section of form(s) and place the form(s) into cooler;
- Pack cooler with ice, as needed, for samples requiring preservation to 4° Celsius (C);
- Place chain-of-custody seals on the exterior of the cooler; and
- Package and ship the cooler to the laboratory.

The following steps will be made upon receipt of the cooler at the subcontract laboratory:

- Inspection of contents;
- Complete requested analyses; and
- Transmit a copy of the original chain-of-custody form(s) with final analytical results from laboratory.

5.2.7 Corrections to Documentation

Original information and data in field logbooks, on sample labels, on chain-of-custody forms, and on any other project-related documentation will be recorded in blue or black waterproof ink and in a completely legible manner. Errors made on any record document will be corrected by crossing out the error and entering the correct information or data. An error discovered on a document will be corrected by the individual responsible for the entry, as possible. Erroneous information or data will be corrected in a manner that will not obliterate the original entry, and corrections will be initialed and dated by the individual responsible for the entry.

5.3 Sample Packaging and Shipping

Sample containers destined for offsite laboratory analysis will be packaged in thermally insulated rigid-body coolers, and will be stored in a secure area during the time period between collection and shipment to the offsite subcontract laboratory. These samples will be packaged, classified, labeled, stored, shipped and tracked in accordance with current US Department of Transportation (DOT) regulations (e.g., 49 CFR 173 et. seq.).

5.4 Management and Retention of Records

Original copies of field data, field records, analytical data, training records, and other project-specific documentation will be retained by the COGCC staff.

6.0 Laboratory Analysis

The contracted laboratory shall perform radiological analysis of solid and aqueous samples for characterization.

Table 2 defines the desired detection limits for laboratory analysis. Table 3 defines the EPA methods applied to the various samples. Table 4 summarizes sample collection, preservative, and holding time requirements for each applicable media on this project. Laboratory analysis of matrix spike/matrix spike duplicate (MS/MSD), field duplicate, and QA split samples will be performed in accordance with the requirements of Section 7 (Quality Assurance).

Samples will be transported to off-site laboratories for analyses in accordance with documented laboratory-specific standard methods listed in the Analysis Methods column of Table 3. Specific sample and laboratory requirements are provided in Section 8.

6.1 Spectroscopic Energy Lines

Radiological COCs for these samples may be quantified for activity concentrations directly via gamma decays, or inferred via gamma-emitting progeny, assuming a secular equilibrium state. Table 2 lists gamma and X-ray emissions from site radiological COCs that may be used for determining soil activity concentrations. The list is broken down into direct emissions from the radiological COC itself or from its decay progeny, which can be used to infer the parent's activity.

Table 2. Approximate Minimum Detectable Activity Using Spectroscopic Gamma Analysis for Radiological Contaminants of Concern

Contaminant of Concern	Direct or Inferred	Inferred Nuclide	Photon Energy (keV)	Yield (%)	Sample MDA (a) Using HPGE (pCi/g)
Ra-228	Inferred	Ac-228	911.2	25.8	1
Th-230	Direct	NA	67.6	0.38	~25 to 125
Ra-226	Direct	NA	186.2	3.59	1 to 5
Ra-226	Inferred	Bi-214	1764.5	46.3	0.1-0.5
Ra-226	Inferred	Pb-214	295.2 and 351.9	15.8	0.1-0.5
U-238 **(b)**	Inferred	Th-234	63.3	4.8	1.9 to 3.5
U-238 **(b)**	Inferred	Pa-234m	1001.0	0.84	~50

Notes for Table 2:

a. The nuclide minimum detection activity (MDA) values stated in the table are from samples analyzed by the HPGe spectrometer in a shielded 1 L Marinelli beaker which is counted for 15 minutes.
b. XRF may be used for solid matrix uranium determination and ICP-MS used for liquid samples.

Table 3. Summary of Sample Types and Laboratory Analysis

Sample Type	Media	Analytical Parameters	Analysis Methods
Drill cuttings	Soil-like	Gamma spec for NORM	USEPA 901.1 or similar
Sediments from produced water	Soil-like	Gamma spec for NORM	USEPA 901.1 or similar
Produced water	Aqueous (grab)	Gross alpha and beta	USEPA 900.0
Produced water	Aqueous (grab)	Gamma spec for NORM	USEPA 901.1 or similar
Produced water	Aqueous (grab)	Liquid scintillation for Rn	Per Lab SOP
Produced water	Aqueous (grab)	Alpha spectroscopy for Pb-210 and Po210	Per Lab SOP

Ra-226 may be measured directly by detection of its 186.2 kilo-electron volt (keV) energy line for high activity waste-sludge samples. However, the presence of U-235 can cause interference with direct Ra-226 detection since it has a gamma line of similar energy (185.7 keV). The short- lived equilibrium daughters of radium may be used to determine Ra-226 concentrations in the soil when background levels of Ra-226 are encountered, for example 1 pCi/g. Once the soil is disturbed, these short-lived daughters must be allowed to grow back in. The parent of these daughters, radon-222 (Rn-222), has a moderate half-life of 3.8 days, therefore requiring at least two to three weeks of progeny in growth to reestablish equilibrium. Gamma spectroscopy will also identify other gamma emitting radionuclides that may be present in samples.

Table 4. Sample Types, Quantity, Container Type, Preservation, and Holding Time.

Parameter	Media	Analysis Method	Sample Quantity	Container Type	Preservative	Holding Time
Gross Alpha and Gross Beta	Solid	USEPA 900.0	20 grams	Plastic	None	None
Ra-226 and Ra-228 by gamma spectroscopy	Solid	USEPA 901.1 or similar	300 grams	Plastic	None	None
Thorium and Uranium Isotopes	Solid	alpha spectroscopy (ASTM D3972)	50 grams	Plastic	None	None
Gross Alpha and Gross Beta	Aqueous	USEPA 900.0	500 ML	Plastic	HNO_3	Up to 6 months
Gamma spectroscopy	Aqueous	USEPA 901.1 or similar	1 Liter	Plastic	HNO_3	Up to 6 months
Total Uranium and Thorium	Aqueous	SW6020	125 ML	Plastic	HNO_3	Up to 6 months
Radon	Aqueous	Per lab SOP	3 X 40ml	Glass	None	3 days
Pb-210	Aqueous	Per lab SOP	1 liter	Plastic	HNO_3	None
Po-210	Aqueous	Per lab SOP	1 liter	Plastic	HNO_3	None

ML - milliliter

7.0 Quality Assurance

The data generated from this SAP must be technically sound, and supported by defined and verified limits of confidence. Therefore, the objective of this Quality Assurance section of the SAP is to ensure the generation of accurate, precise, representative and complete data.

This section provides additional details of the laboratory analytical methods, the quality control of both field instrument s and laboratory equipment, and the quality control program including establishing reference background samples where appropriate, blank analyses, duplicate analyses and spike analyses where possible and applicable. The data collection procedures and data evaluation processes, which will ensure that appropriate levels of data quality are obtained, are also described.

Many of the practices essential to QA, such as use of field logbooks, sample identification, chain-of-custody , packaging, shipping, and management of records have already been described in prior sections and are not repeated here.

7.1 Data Quality Levels

There are typically five analytical levels of data quality, or Data Quality Levels (DQL), available to accomplish the objectives of investigations of this type. These levels are typically designated as follows:

Level I:	field screening or analysis using portable instruments, calibrated to non-compound specific standards;
Level II:	field analysis using portable instruments, calibrated to specific compounds;
Level III:	non-Contract Laboratory Program (non-CLP) laboratory methods;
Level IV:	full record of digestions, cleanups, separations, analytical calibrations, standards used et cetera

The following sections describe the use of the analytical procedural levels for the project.

7.1.1 Field Screening Methods - Level I

The following field screening measurements, described in Section 3.1 will be conducted by COGCC staff. Field screening measurements are defined as Data Quality Level I.

7.1.2 Laboratory Methods - Level IV

Level IV analytical procedures provide precise, accurate, and defensible data for the intended data uses.

7.2 Quality Control Parameters

The exact quantitative criteria used to evaluate data quality from the offsites laboratory's precision and accuracy perspective for the aqueous and solid sampling media will be presented

in the selected laboratory Quality Assurance Manual(s). The following is a description of terms that typically appear in the laboratory Quality Assurance Manual(s).

Reference: The reference identification number of the U.S. EPA or other standard analytical methodology used for each procedure.

Precision: A measure of the mutual agreement among individual measurements of the same property under prescribed similar conditions. Precision is evaluated based on the (DER) between duplicate matrix spike (MS) results or duplicate sample results, as appropriate. The 2 sigma duplicate error ratio (DER) limits are parameter- and method-specific; MS/MSD DER QC limits will be presented in the laboratory QAMs. Laboratory duplicate sample DER limits are typically <2.13 is out of control and >1.42 but <2.13 are in warning range. Field duplicates are also evaluated by calculating the DERs between field duplicate sample results. However, evaluations of field duplicate DERs are used as advisory determinations since numerous factors in sampling and analysis may cause variances between field duplicate results.

Accuracy: The degree of agreement of a measurement with an accepted reference or true value. Accuracy is evaluated based on the percent recovery of spiked samples. The matrix spike recoveries for organic analyses are method- and parameter-specific and are typically used as an advisory QN QC measure due to the difficulty associated with recovering spiked organic parameters. Organic parameter percent recovery QC limits will be presented in the laboratory 's QAM. The matrix spike recoveries for inorganic and most conventional parameters are typically a range of ±25 percent. Chemical and/or isotopic tracer recovery will be evaluated for tests such as determination of radium, uranium and thorium isotopes.

Completeness: A measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under normal conditions. The method of calculation for percent completeness is defined in Section 12.3. Completeness can be evaluated in two ways: 1) by comparing the number of samples actually collected to the expected number of samples to be collected; and 2) by comparing the number of valid analyses received from the laboratory to the number of actual samples collected. The results of any Level III analyses to be

performed are typically used for characterization studies and as such will have a minimum completeness of 95 percent for both evaluations of completeness.

Exact QA/QC criteria the laboratory will use to evaluate its data's precision and accuracy will be provided following selection of the analytical laboratory, if the criteria are not method-specific.

7.3 Calibration Procedures and Frequency

All field and laboratory equipment must be calibrated before use to ensure proper operating capability. Laboratory instrument calibration procedures are presented in the laboratory QAMs. Field calibration procedures and frequencies should be followed in accordance with the manufacture's specifications. Field operational checks must be completed each day at a minimum.

7.4 Preparation of Standards

A calibration standard is prepared by the appropriate dilution of a pure substance, the purity of which is traceable to/National Institute of Standards (NIST) or U.S. EPA standards. Because of the high sensitivity of many analytical instruments, the calibration standard is an extremely dilute version of the pure compound. Because of the high dilution required to be within the linear range of the instrument, the preparation of the calibration standard is frequently made by serial dilution rather than in a single step. In order to provide standard solutions at sufficiently low concentrations, a minuscule amount of the pure substance will be required, the measurement of which is subject to extreme error. Thus, it is preferable to deal with potential dilution errors, rather than with the large error associated with the measurement of a very small amount of the pure substance.

The initial standard is typically obtained either as a pure material or as a prepared certified solution of a given concentration of the pure compound or compounds. In preparing the stock solution of the calibration standard, great care must be exercised in measuring weights and volumes as accurately as possible, since all of the analyses following the calibration will be based on the accuracy of the calibration, and the accuracy of the analytical data is dependent on

the calibration curve. It is the analyst's responsibility to assure that all standards used are within the standard solution holding time, and to prepare fresh standard solutions whenever necessary. In preparing working solutions, or using working solutions, the analyst must check for signs of deterioration of the standard, such as cloudiness, precipitation or discoloration. The standard must also be periodically compared with previous runs of standards, and with independently prepared standards to assure that response factors fall within a historically accepted range.

7.5 Data Evaluation and Validation

Data are typically validated by the field personnel and laboratory personnel. First, during the field operations, field measures will be validated at the time of collection by the field sampler by verifying the use of standard operating procedures for the sampling effort and using field QC checks. Second, laboratory analytical results will be validated by the Laboratory Department Manager or the analyst who is the specific analytical task leader.

7.5.1 Field Data Validation

Validation of field obtained data, as well as ongoing QA/QC checks of environmental samples being taken, is performed on field data. All field data are reviewed during the time of collection and second, all data are reviewed by secondary field personnel if multiple personnel are present. Errors in the field logbooks will be corrected by placing a single line through the entry, initialing and dating the correction. If information is added without a correction being necessary, that entry will be initialed and dated to indicate that it was not entered at the original time of data entry.

7.5.2 Laboratory Data Validation

The individual Laboratory Department Managers shall validate all laboratory data prior to reporting. The following typical QA/QC reviews and/or procedures shall be used:

- Standard calibration curves are prepared prior to sample analysis;
- The standard regression coefficient is within the acceptable range;

- Standard reference materials are analyzed at the proper frequencies and acceptable results are obtained;
- The reagent blanks are analyzed at the proper frequency;
- Precision requirements of this plan are met;
- Accuracy requirements of this plan are met;
- Completeness requirements of this plan are met;
- Samples are analyzed within the proper sample holding times;
- All calculations are verified as correct;
- Proper units are reported; and
- The proper methodologies were used.

In addition to this review of analytical results and project specific precision, accuracy, and completeness requirements, the Quality Assurance officer should perform unannounced audits of report forms and other data sheets as well as regular reviews of instrument logs, performance test results, and analysts ' performance. In the event that any review of analytical results or internal QA/QC checks indicate problems, immediate corrective actions must be taken and all data collected because the previous approved QC audits must be reviewed for validity. Specific laboratory procedures for validation of the analytical data generated are described in the laboratory QAMs.

7.5.3 Data Validation

The laboratory will provide DQO Level IV data packages.

7.6 Data Reporting

After the data have been validated internally by the laboratory, all of the results are electronically or automatically entered into the laboratory's data management system where they are stored prior to reporting. When all analyses are completed, the Laboratory Director (or his/her designee) will issue a final data report including a descriptive case narrative. He or she will then issue the report to the data user.

The data reports generated for this project should contain all pertinent information for the data user to determine the applicability and usability of the data for its intended purposes. For this reason, a specified and uniform data reporting format should be implemented. The following

criteria and information should be supplied, at a minimum, for data reports generated for this project:

- A descriptive case narrative identifying any problems encountered during internal data validation (as described above);
- Completed and legible chains-of-custody for all analyses contained within each submitted data package;
- A lab sample chronicle indicating which analyses were requested and performed for the samples contained in the data package;
- A summary of the laboratory sample identifications and the correlating field sample identifications;
- A summary of all applicable analytical results, errors, MDCs reported in the correct number of significant figures, reporting units; and
- Included in the individual sample reporting results should be the complete sample identifications, the sample dilutions (if necessary), and the individual sample analysis dates.

7.6.1 Level I Reporting

Summary reporting only will be provided. Bulleted items above are required under this Data Quality Level.

7.6.2 Level IV Reporting

The following summary forms and raw data deliverable requirements will apply for Data Quality Level IV.

The following forms are required to be made available (upon request) for all analyses using Gamma Spectroscopy, isotopic Uranium and Thorium, and Alpha Spectroscopy methods:

- Narrative and sample identification cross reference;
- Copies of Chain-of-Custody documentation;
- Laboratory chronicle;
- Method summaries and references;
- Matrix spike/Matrix spike duplicate summary or any lab duplicate;
- QC Check Sample summary;
- Method blank summary and results;
- Instrument performance check summary;
- Instrument set up and calibration summary;
- Continuing calibration check summary for all constituents of interest.

7.7 Quality Control Procedures

Quality control (QC) procedures and checks are used to verify the accuracy of investigation data. Field QC checks are used to identify potential problems with sampling procedures such as the inconsistent use of sampling standard operating procedures or field introduced sample or water supply contamination and/or problems with sample homogeneity or representativeness. Laboratory QC checks are used to identify potential problems with analytical procedures such as the misapplication of required analytical methodologies or other laboratory-related problems which could result in inaccurate or imprecise data reported. The laboratory QC checks and procedures presented in this section are required for most of the applicable methods, but the frequency of the QC checks should follow procedures outlined in the laboratory QAMs.

7.7.1 Field QC

To check the quality of data from field sampling efforts, field duplicate samples will be collected for analysis at a blind duplicate frequency of 1 per ten samples. These samples will be treated as separate samples for identification, logging and shipping. Analytical results on duplicates will be reported with the appropriate field sample data.

7.7.2 Internal Laboratory QC Checks

The QC check frequencies and requirements specified in the following sections is a general description only. The laboratory will follow the internal QC checks specified in its QAM for each analysis type employed. However, these QC checks must meet, at a minimum, the requirements specified in the respective U.S. EPA analytical methods.

The following internal laboratory QC checks are performed for most analyses, whenever applicable, to ensure the measurement systems are under control:

- Initial and continuing calibrations;
- Preparation/method blanks; and
- Matrix spike and matrix spike duplicate or matrix spike and laboratory duplicate analysis, as appropriate.

Additional internal laboratory QC checks are typically performed for most analyses, as required by the associated analytical method. Only the most common QC checks are generally described below.

7.7.2.1 Initial and Continuing Calibration

Each measurement system may be calibrated immediately prior to use and be shown to maintain the calibration throughout the course of the analysis, as appropriate. An initial calibration will be performed and/or confirmed prior to the sample analyses. Continuing calibrations will be typically analyzed at a minimum frequency as recommended by manufacturer and as required in the laboratory's quality assurance plan (LQAP) and standard operating procedures (SOPs).

7.7.2.2 Calibration Check Compounds and Reagent Blanks

Calibration check compounds and reagent blanks are analyzed periodically throughout the course of the analysis, depending upon the required analysis. The exact frequencies and methods of use are presented in the laboratory QAM.

7.8 Performance and System Audits

Two types of audit procedures may be conducted during any environmental investigation: performance audits and system audits. These audits may be performed on the laboratory as well as field activities. A description of the laboratory's specific guidance for Performance and System Audits will be presented in the laboratory QAM. General procedures for laboratory performance and system audits are presented below.

7.8.1 Laboratory Performance Audits

Laboratory performance audits are typically conducted by the Laboratory QA Officer on a routine basis. Each laboratory analyst is provided a performance evaluation sample containing analytes for the parameters which he/she performs. These audit samples are used to identify

problems in sample preparation or analytical techniques or methodologies which could lead to future analytical problems.

Additionally, the laboratory performance audits include verification of each analyst's record keeping, proper use and understanding of procedures, and performance documentation. Corrective action will be taken for any deficiencies noted during the audit.

7.8.2 Laboratory System Audits

Laboratory system audits are typically conducted by the Laboratory QA Officer. These audits are used to ensure that all aspects of the Laboratory's QAM are operative. This involves a thorough review of all laboratory methods performed and documentation to confirm that work is performed according to project specifications.

In some cases, outside certification agencies conduct performance and system audits to verify contract compliance or the laboratories' ability to meet certification requirements on methods of analysis and documentation. Results of these outside certification audits may be reviewed at any time as a check on the laboratory's internal auditing procedures.

7.9 Assessment Procedures for Data Acceptability

The following discussion describes the procedures that will be employed to evaluate the precision, accuracy, and completeness of the generated data.

7.9.1 Precision

Precision is a measure of agreement among individual measurements of the same property under prescribed similar conditions. Precision is assessed by calculating the relative percent difference (RPD) of replicate spike samples or replicate sample analyses according to the following equation:

$$RPD = \frac{R_1 + R_2}{(R_1 + R_2)/2}\ x\ 100$$

Where: R 1 = result 1
R1= result 2

7.9.2 Accuracy[PG6]

Accuracy is a measure of the closeness of an individual measurement to the true value. Accuracy is measured by calculating the percent recovery (%R) of known levels of spike compounds as follows:

7.9.3 Percent Recovery:

$$\%R = \frac{spike\ sample - unspiked\ sample}{spike\ added}\ x\ 100$$

Where: for example "spike added" denotes a concentration

7.9.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system, expressed as a percentage of the number of valid measurements that should have been collected. As is specified in Section 4.2, more than one completeness check can be evaluated. It is calculated as follows:

$$Completeness\ (\%) = \frac{number\ of\ valid\ samples\ reported}{total\ number\ of\ samples\ analyzed}$$

7.9.5 Quality-Control Charts

Quality control charts can be prepared after the initial 20 analytical determinations to graphically evaluate precision and accuracy criteria. The charts are prepared by calculating the mean value of the determinations and setting control limits at ± 3 standard deviations from that mean. Standard defiitions for mean and standard deviations apply and are not shown here.

The control limits should be within acceptance limits or ranges presented in the laboratory's QAM. If the values are found to be outside these limits or ranges, the measurement system is examined to determine if possible problems exist.

7.10 Preventive Maintenance

Periodic preventive maintenance is required for equipment whose performance can affect result. Instrument manuals are kept on file for reference if equipment needs repair. Troubleshooting sections of manuals are often useful in assisting personnel in performing maintenance tasks.

7.10.1 Field Equipment

Field sampling personnel will be responsible for preventive maintenance of all field instruments. The field sampling personnel will protect the instruments by placing them in portable boxes and/or protective cases.

All field equipment will be subject to a routine maintenance program, prior to and after each use. The routine maintenance program for each piece of equipment will be in accordance with the manufacturer's operations and maintenance manual. All equipment will be cleaned and checked for integrity before and after each use. Necessary repairs will be performed immediately after any defects are observed, and before the equipment is used again.

Equipment parts with a limited life (such as batteries, membranes and some electronic components) will be periodically checked and replaced or recharged as necessary according to the manufacturer's specifications.

Preventive maintenance provides for a longer useful life of the equipment and helps to ensure a successful field sampling and testing program. Each piece of field equipment will have its own log sheet which contains the equipment identification and the type of maintenance performed.

Since most equipment is used on an irregular basis, all equipment will be properly stored when not in use.

7.10.2 Laboratory Instruments

All major laboratory instruments should normally be under service contract so that trained professionals are available on call to minimize instrument downtime. Other preventive maintenance schedules and/or procedures for laboratory equipment are presented in the laboratory QAM.

7.11 Corrective Action

There are many laboratory functions that may require corrective action. The decision to undertake corrective action and the ensuing action must be documented so that traceability can be maintained. Corrective action procedures are divided into two subgroups: methods corrective action and systems corrective action. These corrective actions are implemented whenever system or performance audits note deficiencies or when QC procedures indicate a potential analytical problem. The point of originating the corrective action varies, depending upon the mode of detection that such action is necessary. It is generally the role of either the Laboratory QA Officer or the Laboratory Department Manager to initiate such action. Those actions that affect the quality of the data will be recorded and the record maintained by the Laboratory QA Officer. The general procedures for appropriate laboratory corrective actions and identification of potential problems are presented in the analytical laboratory QAM.

7.12 QA Reports to Management

Audit reports will be provided by the Laboratory Director (or his/her designee) as a means of tracking program performance, as applicable, or if needed. Additionally, periodic assessments of measurement data accuracy, precision, completeness and significant QA/QC problems will be performed and reported to laboratory and/or project management, if needed.

Field QA reports will not be necessary considering the expected size and length of any individual sample collection activity. Any problems noted during sampling will be immediately communicated to the COGCC staff member in charge.

The final project report prepared as a result of this SAP should address the overall degree of project conformance to specifications and the impact of any non-conformances that may affect management decisions.

The final storage location of the files will be maintained by the COGCC staff as necessary.

8.0 References

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