Energy Resources Program Geochemical Laboratory Review- Final Report

Laboratory Review Coordination Team
USGS Central and Eastern Energy Resources Science Centers

Introduction

During 2009, a working group within the USGS Energy Resources Program (ERP) conducted a comprehensive review of ERP laboratory functions and quality assurance/quality control (QA/QC) procedures. Members of the laboratory review coordination team were Chris Potter, Jim Coleman, Jamey McCord, Paul Lillis, Terry Lerch, Sue Tewalt, and Doug Duncan. Jim Luppens also participated in the latter stages of the process.

Background

The USGS Energy Resources Program (ERP) Geochemical Laboratories identified significant data quality problems during 2008 that had resulted from inappropriate procedures used by an inorganic chemist employed in the Denver laboratory from 1996 to 2008. The inappropriate procedures included over-use of data normalization techniques coupled with incomplete documentation of raw chemical data generated in the lab. The problems were primarily associated with trace element analyses run on an Inductively-Coupled Plasma Mass Spectrometer (ICP-MS), and also affected some of the single-element inorganic analyses that were run during this period by this same chemist. These issues were serious enough that the overall integrity of the laboratory data was questioned, and fundamental questions were raised with respect to the future mission and directions of the ERP Geochemistry Laboratory Project as well as other geochemical labs funded by ERP. While the laboratory review focused on the labs run under the auspices of the ERP Geochemical Laboratory Project in Denver, the process considered the functions of all geochemistry labs in the Reston and Denver Energy Resources Science Centers.

In addition to the laboratory review steps described below, the review coordination team composed a statement entitled "U.S. Geological Survey Energy Resources Program Geochemistry Laboratory: Data Quality from 1996 through 2008" intended for release to scientific journals in which Energy resources program laboratory data from this period have been published. This statement, which has been reviewed by ERP management, the USGS Ethics Office, Director's Office, and Office of Communications, and the Department of Interior Solicitor's Office, is included as Appendix 1.

Expected outcomes, as defined at the outset of this lab review:

(1) Definition of the mission and goals of the ERP laboratories;

(2) Thorough, objective evaluation of the QA/QC procedures in place in the labs.

(3) Development of a general workflow (and training plan, as necessary) that will emphasize timeliness, accountability, and data archiving/preservation and retrievability within the context of formalized QA/QC procedures.

(4) Assessment of scope and scale of future analytical needs of the ERP projects;

(5) Determination of the specific analyses and functions that will be performed in the ERP labs;

(6) Recommendations for analyses that will be sent to outside labs (and discontinued in ERP labs);

(7) Development of a general review procedure and schedule that will determine on an annual basis if the labs are meeting the research and assessment needs and quality standards of the ERP.

Components of the Laboratory Review

As the laboratory review progressed, outcome goals (2) through (6) were identified as the core of the review process. Outcome goals (1) and (7) are fundamental organizational needs that are referred to Energy Science Center Directors and future lab management for completion.

To address the core outcome goals, the laboratory review coordination team agreed to accomplish:

(1) Cost/benefit analysis of analytical procedures performed in ERP laboratories.

(2) Solicitation of specific feedback for the lab review coordination team, through an internal questionnaire circulated among USGS Energy Resources scientists.

(3) Review of QA/QC laboratory procedures by an objective, qualified third party.

(4) Visits to ERP geochemical laboratories in Denver and in Reston, by members of the lab review coordination team, in order to familiarize (or re-familiarize) team members with the main ERP laboratory functions.

Overview of the ERP Laboratories

The ERP supports a number of laboratories in both Denver, Colorado, and Reston, Virginia. The Denver laboratories that are operated under the Geochemistry Laboratory project were the focus of the ERP lab review. These are laboratories that have multiple customers and have components that typically follow standard operating procedures (SOPs), where high-quality routine operation is the rule. Several other ERP

labs are considered to be research labs, where innovative techniques are explored and special analyses are performed to address non-routine research goals. Whether considered "routine" or "research" labs, all of the ERP labs support the fundamental research mission of the USGS ERP. Initially the team looked at the entire scope of laboratories in the program. After evaluating the extent to which the different laboratory operations were conducted for research, research with a small proportion of conducting analyses for other customers, or primarily for customers, the decision was made to focus on the inorganic and organic geochemistry laboratories in Denver. This decision was made because (1) There had been a known quality problem at the inorganic lab; (2) It was considered a priority to address issues that affect our customers; (3) The smaller research-based laboratories require that a different approach be taken; and (4) lab review coordination team resources were limited. Accordingly, the team focused on the Denver inorganic and organic laboratories that are operated by the ERP Geochemistry Laboratory Project.

The main analytical components of the Geochemistry Laboratory Project include inorganic geochemistry capabilities, x-ray diffraction (mineralogy) capabilities, and organic geochemistry capabilities. These are organized as three separate tasks in the project organization for FY 2010, but were not broken out as separate tasks in FY 2009 and earlier. All components of the project are subject to a common quality assurance policy, and all data from all components are compiled and served using a Laboratory Information Management System (LIMS).

The inorganic geochemistry task addresses the chemistry of inorganic components of coal, coal by-products (such as ash produced by coal-fired power plants), water and sediment. Analytical instruments and associated analyses include:

- Inductively-Coupled Plasma (ICP) for major elements.
- Inductively-Coupled Plasma Mass Spectrometer (ICP-MS) for trace elements.
- Flow-injection Atomic Absorption (AA) single element analyses for Mercury (DMA-80, FIMS100) and Selenium (AA-20).
- Single-Element direct read instruments for Chloride (TOX100) and Sulfur (LECO 432).

Associated procedures include:

- Determination of moisture content in coal.
- 525°C and 750°C Ashing.
- Loss on Ignition associated with 525°C and 750°C Ashing.
- Low Temperature Ashing.
- Ash digestion.

The mineralogy task encompasses X-ray diffraction capabilities (principally, Panalytical X-ray diffractometer).

The organic geochemistry task addresses the geochemistry of solid hydrocarbons, petroleum, natural gas, and hydrocarbon source rocks. Capabilities (and instrumentation) include:

- Bulk fractionation of oil or bitumen extract (column chromatography).
- API gravity determination (density meter, hydrometer, or pycnometer).
- Gas chromatography of crude oils, rock extracts, asphaltic tars and fractions thereof. (two gas chromatographs with auto samplers)

- Biomarker analysis of crude oils, rock extracts, asphaltic tars and fractions thereof. (double-focusing magnetic sector mass spectrometer interfaced with a gas chromatograph with auto sampler).
- Elemental analysis of C, H, N, S, and O in whole oils, extracts, tars, and kerogen (combustion elemental analyzer).
- $\delta^{13}C$ stable isotope analyses of specific hydrocarbon compounds in crude oil and bitumen extracts using an Isotope Ratio Mass Spectrometer (IRMS) interfaced with a gas chromatograph (GC) [under development].
- $\delta^{13}C$ stable isotope analyses of solids (elemental analyzer/IRMS).
- $\delta^{13}C$ stable isotope analyses of natural gas (GC/IRMS).
- δD stable isotope analysis of natural gas (GC/IRMS) [under development].
- Gas composition (customized GC with auto sampler).
- Vitrinite reflectance (microscope with microphotometer).
- Bitumen extraction (typically using Soxhlet apparatus).

Research laboratories operated by ERP projects in Denver include a hydrous pyrolysis lab, a sealed-tube pyrolysis lab, and a methanogenesis lab. Research laboratories operated by ERP projects in Reston include coal petrography, environmental trace element, Raman spectrometry, artificial maturation, aqueous environmental geochemistry, microbiological and organic geochemistry laboratories. These labs are not part of the Geochemistry Laboratory project, so are not included in the focus of this laboratory review process.

Quality Assurance / Quality Control (QA/QC) Review

Over the past several years, the QA/QC procedures of the Denver laboratories have been the subject of laboratory audits performed by Quality Associated International Ltd. (QAI), specifically by its principal, Lou Janke. The Denver inorganic geochemistry labs were audited by QAI in 2005 and in September 2009, and the organic geochemistry labs were audited by QAI in 2007.

In April 2009, as part of the present lab review process specified in outcome goal (2), the ERP engaged the USGS National Water Quality Laboratory (NWQL) Quality Assurance Section to perform an audit to evaluate the effectiveness of the ERP laboratories' quality system and the implementation of a Quality Control Manual (QCM). The ERP laboratory QCM and the NWQL audit report are included herein as Appendices 2 and 3 (respectively). The audit included a review of the QCM and various SOPs. On-site activities included laboratory walk-through, interview of laboratory personnel and review of additional support documentation such as logbooks, training records, and sample tracking systems. The audit report states that analysts are very knowledgeable and communicate openly with auditors. Although the ERP "quality system" is "in its infancy," the auditors concluded that "full implementation of the QCM will provide the USGS Energy Labs with sufficient documentation and traceability to support the quality of data produced." The Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (inorganic) and the Gas Chromatography-Mass Spectrometry (GC-MS) (organic) analyses were considered to be exemplary "representations of how the implementation of QCM requirements should be implemented." The LIMS was found to be "an enormous

asset that facilitates all QA/QC and sample tracking." Overall, the auditors were encouraged by, and complimentary of, the efforts of the Energy labs to implement a quality system. They expressed several concerns, including the need for management support of implementation of the QCM; the need for a QA/QC Manager "to be independent of other positions, so as not to provide a conflict of interest;" the need for reduction of sample backlog, and "requirements for laboratory notebooks that need to be implemented labwide."

Between 2005 and 2008, the Denver laboratories within the Geochemistry Laboratory project had been working toward increasingly rigorous QA/QC procedures, but there was not a formalized guiding document for these procedures. A significant positive outcome of the lab audit process has been the requirement to formalize QA/QC procedures for the Denver labs. The QCM, and the underlying SOPs that apply to each significant procedure performed by the Denver inorganic and organic geochemistry labs, were developed in 2009 and were scrutinized as part of the NWQL audit. The QCM and SOPs are available from the USGS ERP Geochemistry labs in Denver, and from the office of the Energy Resources Science Center in Denver. SOPs can be found at http://energy.cr.usgs.gov/gg/geochemlab/methodology.html.

Although past QA/QC reviews have focused on the Denver labs, the review coordination team agreed that these quality principles and systems should be extended and applied through all ERP-funded geochemistry labs, to the extent that this is practical.

Questionnaire

To meet outcome goal (4), and in part outcome goals 5 and 6, questionnaires were emailed to all scientists in the Denver and Reston Energy Resources Science Centers, along with a report on the results of the NWQL audit. The questionnaires were designed to solicit feedback on several issues, including: (1) the projected volume of USGS ERP scientists' future needs for geochemical analyses of oil, gas, rock, coal, ash, and other sample types; (2) scientists' recommendations with respect to the utility of the specific suite of analyses currently being performed in the Denver labs, and on additional types of analyses that the ERP labs should consider providing; (3) the likelihood of ERP scientists submitting future samples to the labs in Denver and in Reston; and (4) comments from ERP scientists on any other lab-related issues. The questionnaire form and a summary of the questionnaire results are included as Appendix 4.

Twenty-five ERP-funded scientists returned the questionnaire forms. The lab review coordination committee concluded that the majority of the scientists who use the labs responded. A compilation of the responses indicates that USGS Energy scientists expect to submit about 300 oil samples, 550 gas samples, 780 rock samples and 950 ash and coal samples on an annual basis. Based on recent history, lab management considers these numbers to be minimum figures. Nearly every respondent indicated a willingness to submit samples to the Denver organic and inorganic labs in the future, with a few people qualifying their responses with concerns about data quality and timeliness. While most respondents felt the existing suite of analyses offered by the labs are appropriate, a number of respondents expressed a desire for implementation of rare-earth element (REE) analyses, and for the continued development of stable isotope analysis capabilities for natural gas.

With respect to these specific requests for REE and stable isotope analyses: The Denver organic geochemistry lab is working to complete development of its hydrogen/ deuterium and carbon stable isotope analytical capabilities, meeting one of the needs that was strongly expressed in the responses to the questionnaires. In the coming year, Laboratory, Science Center and Program management plan to weigh the costs and benefits of developing the capability to provide REE analyses in coals.

Cost-Benefit analysis

To address aspects of outcome goals (5) and (6), the lab review coordination team assembled a spreadsheet summarizing the costs associated with the analytical capabilities of geochemical facilities housed in the Energy Resources Science Centers in Denver and Reston. This spreadsheet is available in hardcopy form in the offices of the Central and Eastern Energy Resources Science Centers, for review by Energy Resources scientists and staff.

The spreadsheet itemizes each instrument or significant piece of equipment, indicates its laboratory function, and summarizes the amortized capital cost of the instrument/equipment, associated space costs, supply (consumables) costs, and personnel costs associated with the running of this equipment. The average number of samples per year is estimated for each function, and all of this information is used to calculate a cost per sample. (For the Denver labs, the number of samples per year is based on historical trends, averaged for the past five years; for the Reston labs, this number represents samples run during 2008). These costs are compared against the costs of obtaining these analyses/services from outside sources (based on cost sheets from Humble Geochemical Services, Baseline Resolution, Inc, and Geochemical Testing, Inc., phone conversations with these same companies, and discussions with university x-ray diffraction labs). Nearly every analytical function or sample preparation procedure listed for the Denver labs is determined to be more cost-effective than the outsourcing alternative. In almost all cases, the relative cost of in-house *vs.* outsourced procedures represent double-digit percentage savings. Only two procedures were found to be more expensive to perform in-house: low temperature ashing of coal and oil shale (13% greater cost in-house relative to outsourced cost), and chloride analyses for coal (41% greater in-house).

To capture the total costs of the Denver labs, the costs of maintaining and staffing the LIMS and QA functions, as well as lab management costs, are included in the "rolling up" of overall totals for costs of Denver labs. The overall conclusion of the cost-benefit analysis is that the operations of the Denver labs are cost-effective, with projected analytical throughput costing significantly less than an equivalent set of analyses purchased through an outside lab.

Summary/ Recommendations

With respect to QA/QC ("quality systems") in the ERP Denver geochemical laboratories, (outcome goal 2), this review process has accomplished several goals, in: (1) subjecting the labs to rigorous audits, the conclusions of which have endorsed the overall practices and directions of the labs' quality systems while providing constructive recommendations for future improvements; (2) ensuring that appropriate documentation

is in place to formalize these quality systems; (3) demonstrating (through these audits and documentation) that analysts and management are committed to the continued implementation and growth of established QA/QC protocols.

Regarding the two laboratory audits that occurred in 2009, it is interesting to note that, while both audits provided positive feedback and endorsement of the laboratory QA/QC procedures in place, there is a difference in the language and tone used to describe the progress of the quality system. The USGS NWQL review described our quality system as being in its infancy – a reference to the newness of the Quality Control Manual and Standard Operating Procedure documentation; whereas the Quality Associated International Ltd. (Lou Janke) audit concluded that the analysts' involvement in QA/QC demonstrated tangible progress relative to this same reviewers' previous audits of the Denver labs. These two points of view are both considered to be valid summaries of the state of the quality systems in the Denver labs.

The results of the user survey of ERP scientists demonstrated a qualified confidence in, and commitment to, the Denver laboratories going forward. It is clear that the labs will need to demonstrate a track record of high data quality, transparency and reasonable turnaround times to retain (or in some cases, gain) the confidence of ERP scientists. An immediate outcome of this lab review process should be the adoption of a straightforward transparent policy to ensure reasonable and predictable turnaround times for analyses, and reasonable access to the analytical results and QA data through the LIMS.

As summarized above, the cost-benefit analysis does not argue for wholesale conversion to outsourcing of analyses. The cost-effective laboratory operations argue for retaining all, or nearly all, of the analytical functions in-house. As of March 2010, chloride analyses for coal are not being performed in-house because of shifting laboratory staffing assignments and because of the cost savings (identified above) associated with outsourcing this particular procedure.

The review team did not undertake a quality audit of the Denver and Reston research laboratories. In recognition of the non-standard, research nature of these labs, we do not anticipate applying the same audit process to the research labs. Science Center management and Project Chiefs will work toward ensuring that QA/QC principles are applied to the research labs, in customized ways that are consistent with the research mission of these labs.

In summary, this lab review evaluated the QA/QC protocols, user satisfaction and suggestions, and costs related to the analytical procedures in ERP geochemical laboratories, with specific emphasis on the Denver laboratories operated under the Geochemistry Laboratory project. While the labs have undergone a period of transition as they have formalized quality assurance procedures and SOPs, adopted a new LIMS system, and weathered a major challenge posed by the actions of a former analyst that deviated from accepted laboratory quality practices, it seems clear that the Denver ERP geochemical labs are on a path toward continued improvement, transparency and timeliness. It is recommended that the ERP geochemical laboratories be audited again approximately one year after the audit performed during this review, as recommended by the audit team, to determine if the new QA/QC procedures and management commitment have been implemented.

Future success of the ERP laboratories will be possible only if Science Center and laboratory managers are committed to ensuring continued high performance. In addition, the spectrum of analytical procedures and data suites offered should be periodically evaluated and modified if necessary to address the evolving needs of the USGS Energy Resources Program. Finally, it is essential that there is meaningful and professional communication between the laboratory staff and Energy scientists who submit samples to the lab, ensuring that quality issues and related analytical considerations are well-understood and "owned" by lab staff and its principal customers.

Appendix 1. Statement on "U.S. Geological Survey Energy Resources Program Geochemistry Laboratory: Data Quality from 1996 through 2008."
Appendix 2. ERP laboratory Quality Control manual (QCM).
Appendix 3. Summary report of the USGS NWQL audit of quality systems in ERP Denver geochemical labs.
Appendix 4. Questionnaire form (4A) and summary of responses (4B).

APPENDIX 1. Statement released by the USGS to two professional journals that published papers by USGS authors containing data that were potentially affected by past data quality problems.

U.S. Geological Survey Energy Resources Program Geochemistry Laboratory: Data Quality from 1996 through 2008

The U.S. Geological Survey (USGS) Energy Resources Program (ERP) Geochemistry Laboratory initiated a review of its quality assurance practices in 2008, covering quality control and methodology used in inorganic chemical analyses of coal, coal power plant ash, water, and sediment samples.

This quality control review found that inorganic chemical analyses performed by the USGS ERP Geochemistry Laboratory from 1996 through 2008 incorporated quality practices that did not meet standards commonly in use at the time. The most serious shortcoming was the adjustment of raw data to a standard when the instrument reading for the standard was beyond acceptable limits, or when the frequency of repeat analyses of standards was insufficient. In general, adjustment of raw data to account for instrument drift is acceptable practice within strictly defined limits. During the period in question, the maximum adjustment of instrument readings, guided by calibration standards, was not allowed to exceed 10%. However, in some cases the adjustment exceeded 10% and/or was not constrained by an adequate number of control standards. Original instrument readings no longer exist for about 80% of the analyses in question and we are unable to determine the acceptability of drift corrections for most of the samples analyzed during this period. For these reasons, 1996-2008 data from the USGS ERP Inorganic Geochemistry Laboratory should be described as "semi-quantitative" and should be used with care.

For detailed information please visit the USGS Energy Geochemistry Web site (http://energy.cr.usgs.gov/gg/geochemlab/). At that site, a link is available to a list of data sets that have been reviewed by laboratory management and annotated with respect to data quality, and, if applicable, the availability of updated data sets. In addition, individuals may contact laboratory management at EnergyLabs@usgs.gov with specific questions about particular data sets. In some cases, previously analyzed samples remain available, and could be re-run upon request.

APPENDIX 2. Energy Geochemistry Laboratory (EGL) Procedures and QA/QC Manual.

Energy Geochemistry Laboratory (EGL)

Procedures

And

QA/QC Manual ver. 1.2

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Abbreviations

>	Greater Than
<	Less Than
ASTM	American Society for Testing and Materials
CANSPEX	Coal and Ash Sample Proficiency Exchange
CCV	Continuing Calibration Verification
DOI	Department of the Interior
E	Estimated Code
EGL	Energy Geochemistry Laboratory
FOIA	Freedom of Information Act
FY	Fiscal Year
GALP	Good Automated Laboratory Practices
ID	Identification
IT	Information Technology
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LT–MDL	Long-Term Method Detection Level
MDL	Method Detection Limit
MRL	Minimum Reporting Level
MSDS	Material Safety Data Sheet
NARA	National Archives and Records Administration
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
QA/QC	Quality Assurance/Quality Control
SOP	Standard Operating Procedure
SRS	Standard Reference Sample
SRM	Standard Reference Material
USGS	United States Geological Survey

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Manual Overview

This manual gives an overview of U.S. Geological Survey (USGS) Energy Geochemistry Laboratory (EGL) Quality Assurance/Quality Control (QA/QC) and other lab related procedures. Individual Standard Operating Procedures (SOPs) contain the most up to date and detailed information regarding analytical procedures and QA/QC.

Section One: Laboratory Management

Management Roles and Responsibilities

Science Center Director

Responsible for all operations within the Laboratory but delegates responsibilities as appropriate

Project Chief

Manages financial allocations and provides overall direction and supervision

Organic and Inorganic Lab Leads

Works with lab staff to facilitate overall day-to-day operations

Laboratory Information Management System (LIMS) Manager

Oversees LIMS functionality, data integrity, and accessibility

Quality Assurance/Quality Control (QA/QC) Manager

Responsible for overall quality and precision of laboratory data

Quality Assurance/Quality Control (QA/QC) Overview

The USGS EGL uses a three-tiered approach to QA/QC. (1) The first tier is analytical performance based on QA/QC samples analyzed by lab personnel. At this level, lab personnel use the QC sample data to monitor the quality of the analytical process for each batch of samples. If the acceptance criteria in the SOPs are not met, the analysis is discontinued until corrected. (2) The second tier is data review and blind sample programs, where lab personnel use data from these programs to monitor method performance and for long term tracking. (3) The third tier is performance-evaluation studies. These studies can be implemented by private labs, local, State, and Federal agencies, all external to the USGS EGL. Data from these studies can be used to compare laboratories or to select a particular laboratory for analytical work.

Summary reports from many of these studies are available on the EGL web page at http://energy.cr.usgs.gov/gg/geochemlab/.

First Tier: Analytical Performance

Energy lab personnel use QA/QC samples to assess two areas of analytical performance: instrument and method. Accurate instrument operation can be verified by analyzing known standards that have known concentrations or response factors. A blank, which has negligible concentrations of the constituents of interest, is also analyzed to evaluate and eliminate potential sources of bias. In addition to blank samples, standard reference materials are the primary tools for evaluating method bias and variability. Laboratory replicate data are also used to evaluate and obtain method variability information.

Standard Reference Materials (SRMs): SRMs are obtained through internal and external sources such as NIST, CANSPEX, USGS, and EPA. The SRM is thought to be sufficiently tested to be useful in assessing method performance. Several SRMs of differing concentrations and elements are analyzed alongside everyday samples. The SRM data are evaluated to assess bias for each analysis. Additionally, as the data are compiled, long-term estimates of method variability can be obtained.

Laboratory Replicate (Duplicate): The sample to be analyzed is split to provide information about reproducibility and method performance.

Second Tier: Data Review and Blind Sample Programs

Data Review Program: Energy lab analytical results are stored in the LIMS data base. A QA/QC check is done by the QA/QC manager to review all analytical results in the data base. Examples of QA/QC checks include (1) ensuring that QA/QC runs (duplicates, blanks, standards, standard reference materials) fall within acceptable limits (2) checking for the use of internal blind samples, (3) checking historical trends of standards , and (4) checking that correlation coefficients are within acceptable limits (if applicable).

Blind Sample Programs: The blind sample programs are administered by internal EGL personnel and by personnel external to the EGL. The internal program quantifies bias caused by random laboratory contamination. The external blind sample checks method performance and reproducibility.

Third Tier: Performance-Evaluation Studies

The EGL participates in the national and international inter-laboratory performance-evaluation studies such as (1) USGS Branch of Quality Systems Evaluation Program for Standard Reference Samples; (2) CANSPEX; and (3) industry, academia, and Government consortia round robin studies.

Managing Records

The EGL maintains records that (1) describe its management and technical policies; (2) document the procedures that have been followed; and (3) provide support for the technical interpretations, judgments, and discussions concerning laboratory results. These records provide the historical documentation needed for later reviews and analyses.

The storage and retention of records is in compliance with Federal record retention requirements. Records to be maintained are sufficient to reconstruct laboratory activities that produce analytical data. Records

include, but are not limited to, documentation of facilities, equipment, analytical methods, all aspects of sample handling, and data verification.

Records are legible, identifiable, and retrievable and protected against damage, deterioration, or loss.

Records that are stored or produced by computers or personal computers have paper copies or electronic backup copies. These laboratory records typically are maintained near the analytical laboratory or the analyst's workstation until final analysis and data submission.

Types of Records

Types of laboratory records include, but are not limited to, the following:

Standard operating procedures: Any revisions to SOPs are approved by the lab manager and the QA/QC manager. Then they are distributed to all affected individuals to ensure implementation of the changes.

Laboratory notebooks: Provide a record of the analytical methods used and other information pertinent to the EGL analyses.

Calibration records: Provide date of analysis, frequency, conditions, standards, and reference materials. Most such records are stored electronically with the raw data file.

Sample management: Provide a record of sample handling procedures while the sample is in the possession of the EGL. These include records pertaining to (1) sample preservation, including appropriateness of sample container and compliance with holding-time requirement if required in the SOP; (2) sample identification, receipt, acceptance, or rejection and login; (3) sample storage and tracking; (4) sample preparation and analytical request documents; and (5) disposal of hazardous samples.

Sample data: The sample information, methodology, unprocessed data and calculated results for samples are maintained in laboratory notebooks, logs, bench sheets, electronic files, or other sample-tracking or data-entry forms as appropriate for the method. These records may include (1) laboratory sample identification code; (2) date of analysis; (3) instrument identification and instrument operating conditions/characteristics; (4) analytical method used and sample preparation information, including sample aliquots processed, cleanup, separation protocols, and deviations from the SOP; (5) manual, automated, or statistical calculations; (6) confirmatory analytical data, when required to be performed; (7) the analyst's or operator's initials or signature; (8) unprocessed data and calculated results for all QA/QC samples; and (9) source and lot numbers of standards and reagents for traceability. These data and sample information are stored, maintained and retrievable from the Laboratory Information Management System (LIMS).

Correspondence: Correspondence (paper and electronic) for QA/QC and EGL proposals pertinent to a project, generally a written agreement between USGS and EGL management, and communications regarding the progress of the project are kept in accordance with U.S. Geological Survey general records disposition schedule.

Deviations: Deviations from established methods and SOPs are documented in laboratory notebooks and where appropriate documented within the LIMS.

QA/QC Records: QA/QC records include (1) QA/QC summaries, (2) method detection assessments, and (3) in-house audit findings.

Storage and Disposal of Records

Storage and disposal standards for all laboratory records are identified in the General Records Schedule developed in accordance with 44 U.S.C. Chapter 33, *Basic laws and authorities of the National Archives and Records Administration* (NARA) in the U.S. Code of Federal Regulations (U.S. National Archives and Records Administration, 2005).

It is the responsibility of lab management to use the correct disposition to ensure that all records are stored in a secure manner and are easily retrievable. Records disposition schedules are identified in the USGS general records disposition (U.S. Geological Survey, 2003).

Analytical records are kept in the EGL for about 1 year. The records are then boxed, put on pallets, and moved to EGL storage in Building 810, Denver Federal Center. All records are disposed of after a period of 30 years, unless otherwise specified by legal or other written agreements.

As a Federal laboratory, all analytical data produced at the EGL are accessible to the public through the Freedom of Information Act (FOIA). Requests by a third party for access to records must be accompanied by a FOIA request.

Section Two: Quality Control

Data acquired from QA/QC procedures are used to (1) assess the quality of analytical data, (2) determine the need for corrective action in response to identified deficiencies, and (3) interpret results after corrective-action procedures are implemented. Each method SOP includes a QA/QC section that addresses the minimum QA/QC requirements for the procedure. The internal QA/QC checks might differ for each individual procedure, and may include positive and negative controls that are described below.

The application, acceptance limits, and corrective actions for these QC checks are primarily derived from EGL-generated historical analytical data, approved SOPs, and acceptance limits provided by suppliers of materials used to make QC samples. The LIMS allows analysts and management access to QA/QC data very soon after it is generated.

Documentation and Labeling of Standards and Reagents

Records are kept for all commercially-purchased standards and reagents. Records include the manufacturer's Certificate of Analysis or purity (if supplied), purchaser name, and an expiration date after which the material is not to be used unless it is verified.

Original solvent, reagent, reference material, and standard containers provided by a vendor must be labeled with date opened, analyst's initials. An expiration date is the date after which the material may not be used unless confirmed as reliable. Records are maintained for the preparation of standards. These records indicate the source of standard, solvents used, method of preparation, date of preparation, and preparer's initials.

For reagents and standards made on a daily basis, 'daily' may be used in lieu of a specific expiration date on the container and in supporting expiration documentation for those reagents and standards. Date of preparation must still be recorded for all reagents and standards.

In methods where the purity of reagents and solvents is not specified, analytical reagent grade or better is used. Reagents, gases, and solvents of lesser purity than those specified by the method or SOP are not to be used. The labels on the container are checked to verify that the purity of the reagents meets the requirements of the particular method.

Negative Controls

Method blanks: A method blank is an appropriate, well-characterized matrix containing no detectable analyte, that is carried through the entire sample preparation and analytical procedure. The purpose of a method blank is to assess the method for possible contamination during preparation, processing, and instrumental analysis.

For inorganic procedures, the synthetic matrix used generally is the in-house deionized water followed by a final ultrapure deionizing and polishing that results in ASTM Type I reagent. Cartridges to produce the Type I reagent water are replaced as necessary, and cartridge-holders are labeled with the date of installation. For organic procedures, analyte-free organic solvent, organic-free water, or organic-free silica is typically used.

The method blank is processed along with, and under the same conditions as the associated samples to include all steps of the analytical procedure. All reagents are added in the same volumes or same proportions as they are added to the samples. The goal is to have no detectable contaminants; nevertheless, each method blank must be evaluated critically as to the nature of possible contamination and the effects on the analysis of each sample within the batch. If a method blank is contaminated, the source must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank are evaluated as to the best corrective action for the affected samples.

Multicomponent techniques have different procedures for troubleshooting because the remaining sample is too small for analysis or because of lengthy analysis time; these data are qualified in reporting to the customer.

Positive Controls

Laboratory control samples (LCS): The EGL SOP may include one or more of a series of reference samples called LCS. LCS examples include standard reference samples, reagent spikes, certified reference materials, surrogate spikes, and continuing calibration verification (CCV) standards.

The LCS are used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The number of LCS can vary and is either specified in the method or SOP. Data from the LCS are compared to established criteria, and, if found to be outside of the criteria, indicate that the analytical system is out of specification. Relevant LCS data are entered into control charts. Any affected samples associated with an out-of-specification LCS are reanalyzed or the results reported with appropriate data-qualifying codes.

Analyte concentrations in the LCS must be within the calibration range of the analytical methods where possible. LCS that are determined to be within the acceptance criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch.

Samples analyzed along with an LCS that are determined to be "out of acceptance limit" are reprocessed and reanalyzed, or the data are reported with data-qualifying codes. Constituents permanently marked

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with an "E" code (remark code indicating an estimated concentration) are excluded from these requirements because they do not perform ideally in a given method; typically, they cause large standard deviations or frequent performance problems.

For analytical systems that determine many analytes in the LCS, it is statistically likely that some values will be outside the control limits. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (four standard deviations), but within the ME limits. The number of allowable marginal exceedances is based on the number of analytes in the LCS but does not exceed twenty percent of the total analytes in the LCS.

For each method in which marginal exceedance is applied, there must be a written procedure in the method's analytical SOP to monitor the application of ME allowance to LCSs to ensure random behavior, so that persistent systematic problems are not overlooked because of the ME procedure.

Standard Reference Samples and Certified Reference Materials

Standard reference samples (SRS) and certified reference materials (CRM) are purchased from sources outside the EGL such as the USGS, NIST, and CANSPEX. SRS and CRM are used with certificates supplied by the various vendors.

Continuing Calibration Verification Standards

CCV standards are solutions used in instrumental analysis to check instrument responses to the analytes in relation to the calibration curve. CCVs are prepared from the same materials and in the same manner as calibration standards. The concentration of the CCV sample is chosen to allow easy review by the analyst and typically is in the midrange of the calibration curve.

Laboratory Duplicates

Duplicate samples are typically produced in the laboratory for use as laboratory QC samples to check precision during a run.

Method Detection Limits and Levels

Most laboratory reporting levels (LRL) are based on long-term method detection levels (LT–MDL) designed to measure method variations over time. The LT–MDL and LRL data are available to analysts and data users.

For establishing new analytical methods, initial method detection limits are determined by a method detection level (MDL) study. The spike concentration used to conduct the MDL study should be two to five times higher than the final MDL that is determined. This ideal spike concentration is generally difficult to estimate, especially for new instruments. The initial spike concentration can be set at two to five times the instrument detection limit. The MDL initially is determined for the analytes of interest in each method in ASTM Type I reagent water or a matrix appropriate for the method.

MDL or LT–MDL study is not performed for any component for which spiking solutions are not available. For these types of analytes, the MDL is the minimum reporting level (MRL). Because the definition of the MRL is not specific, a MRL can be set at a concentration acceptable to the data user and laboratory as long as a reliable measurement is achieved.

Internal Audits

Internal audits are conducted under the guidance and direction of the QA/QC manager. The QA/QC manager interviews laboratory personnel, observes procedures, and examines records and documentation.

The audits (1) verify that current laboratory activities follow approved SOPs with appropriate QA/QC; (2) ensure that policies and objectives of the EGL QA/QC are documented in SOPs, have been communicated to and are understood by unit personnel, and have been fully integrated into the workflow; (3) provide the means for monitoring data integrity on a yearly basis and examining a sample request from receipt of the sample at the EGL through data release and retrieval by the customer, and (4) ensure that final data entered into the LIMS has supporting raw data.

Audit reports are provided to the Science Center Director. Report responses are documented and tracked by the QA/QC manager.

Corrective Action

Corrective action may be required when a nonconformance or other departure from EGL policies or procedures is identified or data quality is suspect from one or more of the following; (1) results of performance evaluation studies, (2) internal audits, (3) internal and external QA/QC review, or (4) customer complaints.

The goal of corrective action is not only to eliminate such events, but also to reduce repetition of the causes. The identification and resolution of any nonconformance or departure from EGL policies and procedures require analysts and supervisors to; (1) apply the available features in LIMS to review and chart QA/QC data to monitor process performance, (2) treat QA/QC data that are outside the acceptance limits or exhibiting a trend outside control limits as evidence of unacceptable error in the analytical process, (3) initiate prompt corrective action to determine and eliminate the source of the error, (4) hold release of data until the cause of the problem is identified and corrected or qualified, and (5) maintain records of all out-of-specification events, the causes, and the corrective action(s) taken.

If nonconformance is identified, a proposed corrective action is prepared and submitted for approval by the laboratory supervisor or Science Center Director. The proposed corrective action requires an evaluation of the need for action(s) to prevent any recurrence of the problem. The corrective action is based upon a determination of the root cause, assigns responsibility for the action, and includes completion dates.

The QA/QC manager verifies that corrective actions were completed or that the data is qualified. Corrective actions are tracked to determine their effectiveness. Subsequent audit(s) or QA/QC reviews may be performed to confirm that the corrective actions have been implemented and are effective.

Section Three: Training

Data produced by the EGL meet established data-quality objectives, which results from the analyses being performed using good laboratory practices and following relevant SOPs, and the analyst performing the procedure having been properly trained with demonstrated proficiency in the analysis. The EGL provides training and materials (for example, method reference, SOPs, this QA/QC document) and receive training by the supervisor or another experienced analyst.

Initial Demonstration of Proficiency

An initial demonstration of proficiency (IDOP) documents the analyst proficiency in each SOP that they perform. The IDOP is part of the EGL's QA/QC documentation along with ongoing QA/QC such as blind sample and round robin studies that are filed in the QA/QC manager's office.

Ongoing proficiency of the analyst in his/her assigned SOP is maintained by: (1) ensuring that each employee has read, understood, and is using the latest version of the laboratory's in-house SOP, as well as other documentation that relates to his/her job responsibilities; (2) having analysts take new or refresher training courses or workshops on specific equipment, analytical techniques, and laboratory procedures related to their assigned SOPs; and (3) periodic demonstrations of proficiency by the analyst in assigned SOP using laboratory control or blind standards.

Ethics

Data integrity training is mandatory for all analytical personnel. Training records are maintained to ensure that each employee has attended the required training and understands his/her ethical responsibilities and reporting mechanisms. Training for new employees addresses three areas: personal ethics, corporate ethics for Federal employees, and professional ethics that include data integrity.

Professional ethics training includes, but is not limited to (1) data integrity topics such as laboratory fraud and inappropriate laboratory practices (for example, falsifying or fabricating data), misrepresentation of quality-control (QC) or calibration data, improper spiking procedures, improper instrument clock setting/recording, method deviation, and other breaches of ethical behavior; (2) the training covers the need for honesty and full disclosure in reporting; (3) suggests ethically challenging scenarios that give personnel the opportunity to gain insight through discussion; (4) instructs personnel on ethics expectations for business conduct and makes them aware of the effect of inappropriate practices; and (5) ethical and legal responsibilities of Federal and non-Federal employees, including the potential punishments and penalties for improper, unethical, or illegal actions. See Appendix A for USGS Scientific Integrity policy.

The EGL management determines specific data integrity training.

Safety Training

All Federal and non-Federal employees that work in the laboratory receive annual safety training to meet the requirements of the Occupational Safety and Health Act, Colorado Department of Public Health and Environment (1998), and U.S. Geological Survey (2002) requirements. Training includes instruction on the elements of the Occupant Emergency Plan and the Chemical Hygiene Plan, and hazard communication information. Some staff members receive specialized safety training, such as emergency response hazardous material spill containment and cleanup, first aid/cardiopulmonary resuscitation, automated external defibrillator response, and fume hood training. Federal employees who drive on official business must complete defensive-driver training every 3 years.

All new Federal and non-Federal employees receive orientation training in the EGL Safety, Health, and Environmental Compliance Program, generally within the first week of employment.

Section Four: Instrumentation

Initial Calibration

The initial calibration of an instrument prior to use is documented. The documentation includes calculations, integrations, acceptance criteria, associated statistics, and other pertinent information sufficient to reconstruct the calibration. In all cases, the initial calibration is verified (as indicated in the published analytical method or approved method SOP) using an independently prepared (third-party) calibration verification solution other than the one supplied by the manufacturer (unless, as in some organic analyses, this step is not possible).

Continuing Instrument Calibration

All analytical instruments in use for testing are uniquely identified. Continuing calibration procedures for a specific laboratory analytical instrument should consist of a calibration of at least three standards or two calibration verifications throughout each analysis. An exception to this standard is instrument technology that uses only a zero and a single-point standard, such as inductively coupled plasma atomic emission spectrometry. The SOP for each analysis performed describes the calibration procedures, frequency, acceptance criteria, and the conditions that require recalibration (unless, as in some organic analyses, this step is not possible).

All method SOPs provide specific details of instrument calibration and incorporate, where appropriate, the following features: (1) matrix-matched reagent blank used to establish calibration baseline; (2) use of the same preservation matrix for standards and samples; (3) adequate number of standards used to define calibration; (4) use of the low check standard is addressed as appropriate for the method; (5) application of appropriate curve fit; (6) linearity of curve is established if relevant; (7) use of control sample, third-party check where available, to verify calibration accuracy; (8) use of verification standard, continuing calibration verification or reference sample, to verify system stability; (9) acceptance for calibration and process for dropping calibration points; and (10) a sufficient number of unprocessed data records must be retained to permit reconstruction of the initial instrument calibration.

Log books: A log book is maintained for each instrument and analytical system, which can contain, but is not limited to, the following information: instrument identification, serial number, date of calibration, operator, calibration solutions with traceability, and analysis file information. The log books are kept with the instrument for a minimum of 1 year after completion; when they are then transferred to the laboratory records for archiving.

Control charts: QA/QC data are entered into control charts upon completion of analyses. These data are examined by designated staff before release. Suitability of data submission is based on historical or predetermined acceptance criteria. QA/QC data are available for control-charting functions when data are approved in LIMS.

Verification of calibration: All verifications of calibration are traceable to the analytical method, analyst, instrument, analysis date, analyte name and concentration, and response (or response factor). When instrument software allows for this capability, sufficient information is recorded and archived to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method requirements and are documented in the method reports and SOPs. Whenever practicable, equipment requiring calibration is to be labeled to show its calibration status, including the last and the next calibration dates.

Failure of initial calibration: All results are calculated on the basis of the response curve from the initial calibration or from subsequently adjusted analyses. If the initial calibration fails, the analytical procedure is stopped and evaluated. For example, a second standard might be analyzed and evaluated or a new initial calibration curve might be established and verified. In all cases, the initial calibration must be acceptable before reporting data for any affected samples.

Calibration verification checks: When an instrument is not calibrated on the day of analysis, a calibration verification check standard is analyzed at the beginning and at the end of each batch of about 10 samples. The concentration of this calibration check could vary on a regular basis and is described in the method SOP. If a calibration check standard fails, and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria, then a new calibration curve is prepared.

Data calibration: Some SOPs may require that, data not be reported if values are greater than the highest calibration standard used (extrapolated). The sample must be diluted, if possible, so the concentration or response factor falls within the calibration range. The observed concentration or response is then multiplied by the dilution factor.

The only exception to this rule are those constituents that cannot be reanalyzed in which case these data are qualified in the appropriate manner. For concentrations that are reported to be less than the lowest calibration standard, the data also must be appropriately qualified (these two situations are qualified with an estimated or “E” code).

Preventive Maintenance

Log books are maintained for each major piece of equipment, with all reference materials applicable to the analysis performed. These records include documentation on all routine and non-routine maintenance activities. The records may include: (1) the name of the equipment; (2) the manufacturer’s name, type identification, and serial number or other unique identification; (3) the date received and placed into service; (4) the location; (5) the condition when received (for example, new, used, reconditioned); (6) the location of a copy of the manufacturer’s instructions in the laboratory; (7) details of maintenance performed and all plans for preventive maintenance, including dates; (8) a history of any moves, damage, malfunction, modification, or repair of instruments; (9) identification of supporting software; and traceability standards referenced to NIST or other standard source.

A routine preventive maintenance program is used to minimize instrument failure and other system malfunctions. Designated Federal and non-Federal employees, and service contractors regularly perform routine scheduled maintenance and repair. Maintenance is documented in the instrument records.

Failure to meet specified acceptance criteria requires that the equipment be removed from service. Before returning to service, any equipment must be calibrated and the new calibration curves verified and documented.

Section Five: Samples

Sample Tracking

The EGL uniquely identifies each sample for analysis to ensure absolute reliability regarding identity. The sample login system includes individual identification of all samples. Subsamples, subsequent extracts and digestates are tracked by analyst.

A unique identification (ID) code is placed on the sample container. This is referred to as the laboratory ID and consists of the E, the two-digit year, the two-digit month, the three-digit job number, a dash, and the sequential three-digit sample number. (Example, E0903001-005.)

Sample Disposal

Unless samples are returned to the submitter at their request, samples are disposed of in accordance with Federal, State, and local regulations (Colorado Department of Public Health and Environment, 1995). Depending upon the composition of the sample, disposal options include neutralization and passing through the ion exchange system before sewer discharge or shipment to a waste disposal facility that is approved and permitted by the U.S. Environmental Protection Agency.

Section Six: Safety and Health

Safety

The USGS and EGL is committed to providing a safe and healthful environment for Federal and non-Federal employees, contractors, and visitors through a program of compliance with the Occupational Safety and Health Act, Executive Order 12196 (Occupational Safety and Health Programs for Federal Employees), and all applicable Federal, State, and local regulations. The primary basis and requirements for the USGS are in the handbook (U.S. Geological Survey, 2002).

The EGL is responsible and accountable for creating and maintaining a safe working environment through effective health and safety programs, regular inspections and assessments of the workplace, staff training, and guidance and support to safety efforts. All lab staff is responsible for working safely by following established safety and operating rules and procedures, maintaining an awareness of potentially hazardous situations, reporting all unsafe conditions to supervisors, and reporting all accidents or incidents that result in, or could have potentially resulted in, personal injury or property damage.

The safety guidelines apply to all activities and operations of the EGL, as well as the Federal and non-Federal employees, volunteers, contractors, and visitors that it serves. Guidelines include those for (1) development of organizational policy, plans, programs, directives and rules, and interpretation of safety and health policy and procedures including management and personnel accountability, working groups that address safety, health, and environmental issues, and the designation of appropriate personnel and

financial resources for implementation; (2) personnel awareness of, and accessibility to, applicable policy, documents, codes, regulations, and standards; (3) performance of annual evaluations of safety elements, that include personnel and financial resources, to provide management with information on program effort and effectiveness and to establish short- and long-term goals for safety enhancement and implementation; (4) conducting operational and facility surveys, inspections, evaluations, and staff visits for the purpose of identifying hazards within the workplace and determining the level of organizational compliance with standards; (5) hazard identification and abatement for organizational reporting and correction of unsafe or unhealthful working conditions and identification/correction of workplace hazards through job hazard analysis; (6) investigation, reporting, and analyzing accidents and providing necessary corrective actions; (7) identifying, developing, coordinating, scheduling, and conducting required training; (8) providing standard and regulatory compliance assistance, awards and recognition programs, and developing safety and health promotion/awareness plans; (9) performing industrial Hygiene/Occupational Medicine studies to include hearing conservation, respiratory protection, personal protective equipment, and laboratory safety; (10) ensuring that EGL facilities and operations are compliant with established fire safety practices and policies; (11) ensuring that operators of motorized vehicles are identified and trained and that equipment is maintained in safe working condition; and (12) providing equivalent safety protections for non-Federal employees, contractors, concessionaires, volunteers, and visitors.

Medical Surveillance

The EGL provides occupational medical surveillance to Federal and non-Federal employees who are occupationally exposed to hazards (U.S. Geological Survey, 2002). The surveillance program includes baseline, periodic, and exit exams.

Selected References

Colorado Department of Public Health and Environment, 1995, Colorado hazardous waste regulations: 6 CCR 1007-3, November 1995, variable pagination with updated pages.

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National Environmental Laboratory Accreditation Conference, 2001, Quality systems (Chapter 5): accessed October 28, 2005, and archived at URL http://www.epa.gov/nelac/standard/5qs-15-0-corr3.pdf

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Appendix

Appendix A. Scientific Integrity

This text comes directly from chapter 500.25 in the U.S. Geological Survey Manual and has not been modified.

U.S. Geological Survey Manual

500.25- Scientific Integrity

01/05/07

OPR: Office of Human Resources

Instruction: New SM chapter issued to establish the USGS policy for Scientific Integrity.

1. **Purpose.** This chapter establishes USGS policy for ensuring scientific integrity in the conduct of scientific activities and procedures for reporting, investigating, and adjudicating allegations of scientific misconduct by USGS employees and volunteers. Volunteers include all scientists working under Scientist Emeritus agreements.

2. **Scope.** This chapter applies to all USGS employees and volunteers.

3. **Authority.** Authority for this policy is the Federal Policy on Research Misconduct, Office of Science and Technology Policy, Executive Office of the President. References include:

A. Federal Policy on Research Misconduct, 65 Federal Register (FR) 76260-76264, December 6, 2000, (http://www.ostp.gov/html/001207_3.html).

B. Standards of Ethical Conduct for Employees of the Executive Branch, 5 Code of Federal Regulations (C.F.R.) 2635, (http://www.usoge.gov/pages/laws_regs_fedreg_stats/oge_regs/5cfr2635.html).

C. Department of Interior (DOI) Manual chapter on Discipline and Adverse Actions, 370 DM 752, (http://elips.doi.gov/app_DM/act_getfiles.cfm?relnum=3705).

4. **Policy.**

A. *General*. The USGS is dedicated to preserving the integrity of scientific activities conducted by its employees and volunteers. The USGS will ensure that all employees and volunteers

understand their obligation to abide by this policy, including the USGS Code of Scientific Conduct (section 7), and the Federal Policy on Research Misconduct. The USGS will take appropriate action to protect the public from the effects of inaccurate or misleading information produced through scientific activities, and for violations of this Survey Manual chapter or the Federal Policy on Research Misconduct.

B**.** *Privacy and Confidentiality.* The investigation of an allegation of scientific misconduct will be handled in a manner that protects, as much as possible, the rights, privacy, and professional credentials of any USGS employee or volunteer who makes an allegation of scientific misconduct or who is the subject of such allegation. Investigations will be conducted in a fair and timely manner and in accordance with the Federal Policy on Research Misconduct. The Federal Policy on Research Misconduct states that to the extent possible, consistent with a fair and thorough investigation and as allowed by law, knowledge about identity of subjects and informants is limited to those who need to know. Records maintained by the agency during the course of responding to an allegation of research misconduct are exempt from disclosure under the Freedom of Information Act to the extent permitted by law and regulation.

C. *Protection from reprisal for reporting scientific misconduct*. The USGS is committed to ensuring that employees and volunteers who have a reasonable belief that a violation of the USGS Code of Scientific Conduct and/or the Federal Policy on Research Misconduct has occurred and who report such violation in accordance with this Survey Manual chapter will not be subject to reprisal.

D. *Potential disciplinary action against USGS employees.* If scientific misconduct is found to have been committed by a USGS employee, appropriate disciplinary action will be taken against the employee, up to and including removal of the employee from the Federal service. Disciplinary actions against USGS employees will be in accordance with Departmental Manual chapter 370 DM 752.

E. *Potential administrative action against USGS volunteers*. If scientific misconduct is found to have been committed by a USGS volunteer, appropriate action will be taken against the volunteer, up to and including termination of the volunteer agreement.

5. **Definitions.**

Fabrication is making up data or results and recording or reporting them. (Federal Policy on Research Misconduct)

Falsification is manipulating research materials, equipment, or processes, or changing or omitting data or results such that the research is not accurately represented in the research record. (Federal Policy on Research Misconduct)

Plagiarism is the appropriation of another person's ideas, processes, results, or words without giving appropriate credit. (Federal Policy on Research Misconduct)

Research record. The record of data or results that embody the facts resulting from scientific inquiry, and includes, but is not limited to, research proposals, laboratory records, both physical and electronic, progress reports, abstracts, theses, oral presentations, internal reports, and journal articles. (Federal Policy on Research Misconduct)

Scientific activities. Activities involving inventorying, monitoring, experimentation, study, research, modeling, and scientific assessment. Scientific activities are conducted in a manner specified by standard protocols and procedures and include any of the physical, biological, or social sciences as well as engineering and mathematics that employ the scientific method. Inspections for regulatory compliance and resulting records are not included because they are covered by separate requirements.

Scientific assessment. Evaluation of a body of scientific or technical knowledge which typically synthesizes multiple factual inputs, data, models, assumptions, and/or implies best professional judgment to bridge uncertainties in the available information.

Scientific method. A method of research in which a problem is identified, relevant data are gathered, a hypothesis is formulated from these data, and the hypothesis is empirically tested.

Scientific misconduct. Fabrication, falsification, or plagiarism in proposing, performing or reviewing scientific activities and their products.

Scientific product. The results of scientific activities including the synthesis, compilation, or translation of scientific information into formats used in the Department's decision making process.

6. **Responsibilities**.

A. All USGS employees and volunteers must comply with this chapter and the Federal Policy on Research Misconduct.

B. Each Cost Center Chief must ensure that all:

(1) USGS employees and volunteers are provided copies of USGS and Federal policies concerning scientific misconduct; and

(2) Employees and volunteers who are covered by these policies comply with USGS and Federal requirements to maintain scientific integrity and do not engage in fabrication, falsification, plagiarism in proposing, performing, or reviewing scientific activities and their products.

C. Any person with knowledge that an act of scientific misconduct may be planned, is imminent, or has occurred must report it to their supervisor or other appropriate manager or office, such as the Cost Center Chief, servicing human resources office, the USGS Ethics Office, or the DOI Office of Inspector General. In accordance with paragraph 8A(1) of this Chapter, allegations of scientific misconduct against USGS employees and volunteers must be submitted

in writing to the servicing human resources office within 60 days of discovering the alleged misconduct.

7. USGS Code of Scientific Conduct

The USGS employees and volunteers who engage in scientific activities will abide by the Federal Policy on Research Misconduct, 65 Federal Register (FR) 76260-76264, December 6, 2000, (http://www.ostp.gov/html/001207_3.html) and the following Code of Scientific Conduct:

A. I will act in the interest of the advancement of science and contribute the best, highest quality scientific information for the U.S. Geological Survey and the Department of the Interior.

B. I will conduct, process data from, and communicate the results of scientific activities honestly, objectively, thoroughly, and expeditiously.

C. I will be responsible for the resources entrusted to me, including equipment, funds, my time, and my employees' time. I will promptly and accurately collect, use, and report all financial resources under my control; and promptly, thoroughly, and accurately report all scientific work.

D. I will fully disclose all research methods used, available data, final reports, and publications consistent with applicable laws and policy.

E. I will respect, to the fullest extent permitted by law, confidential and proprietary information provided by communities, Indian tribes, and individuals whose interests and resources are studied or affected by scientific activities or the resulting information.

F. I will maintain scientific integrity and will not engage in fabrication, falsification, or plagiarism in proposing, performing or reviewing scientific activities and their products.

G. I will welcome constructive criticism of my scientific activities, will welcome and participate in appropriate peer reviews, and will critique others' work respectfully and objectively. I will substantiate comments that I make with the same care with which I report my own work.

H. I will be diligent in creating, using, preserving, documenting, and maintaining collections and data.

I. I will adhere to established quality assurance and quality control programs.

J. I will follow the Department's records retention policies and comply with Federal law and agreements related to use, security, and release of confidential and proprietary data.

K. I will adhere to appropriate standards for reporting the results of scientific activities and will respect the intellectual property rights of others.

L. I will, to the extent possible and practical, differentiate among facts, opinions, hypotheses, and professional judgment in reporting the results of scientific activities to others, including scientists, decision makers, and the public.

M. I will be responsible for the quality of any data I collect or any interpretations I make, and for the integrity of conclusions I draw in the course of my scientific activities.

N. I will place quality and objectivity of scientific activities and reporting of their results ahead of personal gain or allegiance to individuals or organizations.

8. **Procedures.** A flow chart of the process to be followed in cases of alleged scientific misconduct is contained in Figure 1, *Flow Chart of Actions to be Taken in Cases of Alleged Scientific Misconduct*.

A. *Allegations of scientific misconduct*.

(1) Allegations of scientific misconduct against USGS employees and volunteers must be submitted in writing to the servicing human resources office within 60 days of discovering the alleged misconduct. In cases of fraud, waste, and abuse that are referred to the DOI Office of the Inspector General, the ability to report misconduct is not time limited. Allegations shall contain the following information:

(a) The name, office location, and signature of the person or persons reporting the alleged scientific misconduct.

(b) The name and office location of the person or persons alleged to have committed the alleged scientific misconduct.

(c) A description of the allegation, with as much detail as possible about the nature of the scientific conduct and the circumstances.

(d) Date(s) the alleged scientific misconduct occurred or continues to occur.

(e) Documents and/or other relevant items (such as data, materials, etc.) pertaining to the alleged scientific misconduct.

B. *Inquiry of allegation of scientific misconduct*.

(1) Upon receipt of an allegation of scientific misconduct, the servicing human resources office will contact the immediate supervisor of the subject of the inquiry (henceforth referred to as the subject) to inform them that an allegation of scientific misconduct has been filed.

(2) The servicing human resources office will provide assistance to the supervisor in conducting an inquiry to determine if the allegation is covered under the provisions of this chapter and will provide consistency, oversight, and guidance throughout the entire process. The supervisor will ensure that all original research records and materials relevant to the allegation are immediately

secured. Once the evidence is secure, the subject will be notified in writing (Appendix A, *Notification of Preliminary Investigation*) that an allegation of scientific misconduct has been filed against them. A supervisor should never act alone in determining the merit of an allegation, but must work with the servicing human resources office.

(3) If after review of the information gathered during the inquiry the supervisor determines that an investigation by the Scientific Misconduct Review Panel (SMRP) (Appendix B. *Scientific Misconduct Review Panel*) is not required, no further action will be taken against the subject. The supervisor will issue a memorandum to the subject explaining that no further action will be taken concerning this incident, and that the case will be closed (Appendix C, *Sample Closure Memorandum*). If the subject is a USGS employee, he/she may provide a copy of this memorandum to his/her servicing human resources office for placement on the left hand side (temporary side) of the Official Personnel Folder, where it will be retained for one year, in accordance with the USGS General Records Disposition Schedule 432-1-SI.

(4) If it is determined that an investigation is required, the supervisor will refer the matter to the Chairperson of the SMRP and will inform the subject that the allegation of scientific misconduct will be investigated by the SMRP. At this time, the appropriate Cost Center Chief will be verbally informed of the investigation.

(5) Allegations of scientific misconduct that involve alleged fraud, waste and abuse, or criminal law violations will be referred to the DOI Office of the Inspector General (OIG).

(6) Allegations of administrative, non-scientific misconduct against a USGS employee or volunteer will be handled through normal administrative processes, in accordance with DOI/USGS regulations and union contracts.

C. *Investigation by Scientific Misconduct Review Panel (SMRP)*

(1) The SMRP shall consist of a Chairperson, four additional scientists, and an alternate scientist. The alternate will serve on the panel when it is necessary for one of the panel members to excuse themselves because of a conflict of interest or if a panel member is prevented from participation for some other reason. The Director of the USGS, in consultation with the Discipline Chief Scientists, shall appoint the panel members. Panel members will serve a 4-year term and may serve for no more than two consecutive terms. To ensure panel continuity, half of the originally selected panel members will serve for 2-year terms and half of the originally selected panel members will serve for 4-year terms. Ad hoc panel members may serve as panel members in order to provide necessary technical assistance. They may be chosen from a specific discipline or a specific area of expertise that is not represented on the permanent panel. The Chairperson, with the concurrence of the Director, will select these panel members as needed. The Director may replace any panel member at any time.

(2) The subject of the investigation will be notified by the Chairperson of the Panel that the SMRP will be convened to conduct an investigation of the allegation of scientific misconduct. The subject will be advised of the investigation and his/her rights and responsibilities during this process. Subjects will be asked to sign the *Employee/Volunteer Information and*

*Acknowledgement Form (*Employee form at Appendix D, Volunteer form at Appendix E*)* acknowledging he/she has been notified of the investigation, his/her rights and responsibilities in complying with the investigation, the consequences of not fully complying to the best of his/her abilities, and the opportunity to respond to the allegation and present testimony and evidence to the panel orally and/or in writing. The Chairperson of the Panel shall retain the original of the form and provide a copy of the signed document to the subject of the allegation.

(3) The Panel will conduct an investigation utilizing one or both of the following methods: (a) securing and reviewing documentary evidence, including all original experimental records, protocols, and data; (b) interviewing relevant persons, whether in person or by telephone, and, as necessary, securing written statements from the interested parties. The Panel will provide the subject of the investigation with the opportunity to respond to the allegation and present evidence and testimony to the SMRP.

(4) Three criteria are necessary to establish research misconduct (Federal Policy on Research Misconduct): (1) There is a significant departure from accepted practices of the relevant research community; (2) the misconduct is committed intentionally or knowingly or recklessly; and (3) the allegation is proven by a preponderance of evidence. The Panel will arrive at a consensus decision, if possible. Consensus decision means that all panel members, including the Chairperson, agree with a decision. This is distinct from a majority-rule decision. In the consensus-based process, panel members work together to develop a finding with which all of the members of the panel can agree. The Chairperson will determine if consensus has been reached by asking all panel members if they agree with the finding. If consensus is reached, then the Panel shall write a report of their findings that contains a summary of the findings, the basis for determining whether or not scientific misconduct occurred, and an assessment of the seriousness and extent of any misconduct found that is in violation of the USGS Code of Scientific Conduct.

(5) The Panel will take the time necessary to address all of the relevant issues associated with the allegation in order to reach a consensus finding. If, after all efforts are exhausted, the Panel is still unable to reach consensus about whether or not misconduct has occurred, then a majority decision will be made. Panel members will write majority and minority reports to send to the subject's immediate supervisor and the servicing personnel office.

(6) Within 30 calendar days of the completion of the report, the Chairperson of the Panel shall forward the report to the servicing human resources office and the subject's immediate supervisor for appropriate action.

D. *Administrative Action*. After receiving the report, the servicing human resources office and the immediate supervisor will determine possible corrective action and, if necessary, appropriate disciplinary action/adverse action for the subject in accordance with DOI/USGS policies and /or union contracts, as applicable. Within 30 days of receipt of the report, the servicing human resources office specialist and the immediate supervisor will meet with the subject to discuss any pending disciplinary/adverse action. If disciplinary/adverse action is proposed, the subject will be provided with a written notice containing the following information: the specific nature of the offense, the type of disciplinary/adverse action being proposed, a right to answer (orally, in

writing, or both), the material relied upon regarding the action, and the right to representation. Once a decision is made, a written decision and appropriate appeal rights will be provided to the subject.

If, based on the report issued by the SMRP, no administrative action will be initiated, the subject's supervisor will notify the subject and Cost Center Chief in writing of the finding. A copy of this memo will be included in the record.

E. *Appeal Rights for USGS Employees*. For disciplinary actions up to and including a 14-day suspension, subjects have the right to appeal through the Administrative Grievance procedure or a Negotiated Grievance Procedure (NGP) if the subject is covered by a collective bargaining agreement. For suspensions of more than 14-days or removal from the Federal service, subjects have the right to appeal to the Merit Systems Protection Board or through an applicable NGP.

F. *Reconsideration Rights for Volunteers.* Volunteers have the right to appeal findings of scientific misconduct and associated actions to the Director of the USGS.

G. *Notification of person filing the allegation:* At the conclusion of the process, the person who filed the allegation will be notified by the servicing human resources office that management has taken appropriate action and that the matter has been resolved.

H. *Records.* Servicing human resources offices will submit a report each calendar year to the Bureau Human Resources Officer. The report will list (1) the number of allegations of scientific misconduct filed against employees and against volunteers, (2) whether or not scientific misconduct was found and (3) the disciplinary or other action taken, if any. These reports will be retained in accordance with Section 407 of the U.S. Geological Survey General Records Disposition Schedule 432-1-S1 and the Privacy Act.

Data that serve to document any allegation or finding of scientific misconduct will be retained by the servicing human resource offices in accordance with Section 407 of the U.S. Geological Survey General Records Disposition Schedule 432-1-S1 and the Privacy Act.

Appendix B. Energy Geochemistry Analysis List

Inorganic Analysis

AA_Hg
Hg
AA Se
Se
ASH525
525Ash
ASH750
750Ash
Chloride
Cl
Geochemical Testing
GT AirDryLoss
GT AshFluid
GT AshHemi
GT AshInit
GT AshSoft
GT AstmAsh
GT CalorificValue
GT Carbon
GT EQM
GT FixedCarbon
GT FSI
GT Hydrogen
GT Moisture
GT Nitrogen
GT OrganicSulfur
GT Oxygen
GT PyriticSulfur
GT ResidualMoisture
GT SulfateSulfur
GT Sulfur
GT VolatileMatter
ICP_MajorForSolids
Al2O3
Ba
CaO
Fe2O3
K2O
MgO
Na2O
P2O5
SiO2
SO3
Sr
TiO2
ICPMS_TraceForSolids
As
Be
Bi
Cd
Co
Cr
Cs
Cu
Ga
Ge
Li
Mn
Mo
Ni
Pb
Rb
Sb
Sc
Th
Tl
U
V
Y
Zn
LOI525
525LOI
LOI750
750LOI
MOISTURE
Moisture
Sulfur
S
XRD
USGSXRD

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Organic Analysis

GCFID_LightHC
1,1-DMCP
3-EP
Benzene
c-1,3DMCP
C3-22DM
C4-2,2DM
C4-2,3DM
C5-2,2DM
C5-2,3DM
C5-2,4DM
C5-2M
C5-3M
C5-CYC
C5-MCYC
C6-2M
C6-3M
C6-CYC
C6-MCYC
i-C04
i-C05
n-C04
n-C05
n-C06
n-C07
n-C08
t-1,3DMCP
t-1,4 DMCP
TOLUENE
GCFID_Sat
Beta Carotane
CPI01
CPI02
CPI03
CPI04
i-C13
i-C14
i-C15
i-C16
i-C18
i-C19 (pristane)
i-C20 (phytane)
i-C21
n-C08

n-C09
n-C10
n-C11
n-C12
n-C13
n-C14
n-C15
n-C16
n-C17
n-C18
n-C19
n-C20
n-C21
n-C22
n-C23
n-C24
n-C25
n-C26
n-C27
n-C28
n-C29
n-C30
n-C31
n-C32
n-C33
n-C34
n-C35
n-C36
n-C37
n-C38
n-C39
n-C40
OEP01
OEP02
OEP03
PH18
PR17
PRPH
GCFID_Whoil
C6-MCYC
i-C04
i-C05
i-C13
i-C14
i-C15

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i-C16
i-C18
i-C19 (pristane)
i-C20 (phytane)
i-C21
n-C04
n-C05
n-C06
n-C07
n-C08
n-C09
n-C10
n-C11
n-C12
n-C13
n-C14
n-C15
n-C16
n-C17
n-C18
n-C19
n-C20
n-C21
n-C22
n-C23
n-C24
n-C25
n-C26
n-C27
n-C28
n-C29
n-C30
n-C31
n-C32
n-C33
n-C34
n-C35
n-C36
n-C37
n-C38
n-C39
n-C40
GCMS BIOM
25-Norhopane
5B Cholane
BISHOP_R

Bisnorhopane
C19 Tricyclic
C19C23 R
C20 Triaromatic Steroid
C20 Tricyclic
C21 Monoaromatic Steroid
C21 Triaromatic Steroid
C21 Tricyclic
C22 Monoaromatic Steroid
C22 Tricyclic
C22C21 R
C23 Tricyclic
C24 Tetracyclic
C24 Tricyclic
C24C23 R
C25 22R Tricyclic
C25 22S Tricyclic
C26 20R+C27 20S Triaromatic Steroid
C26 20S Triaromatic Steroid
C26 22R Tricyclic
C26 22S Tricyclic
C26C25_R
C26TET R
C27 17a Tm
C27 18a Ts
C27 20R 5a Monoaromatic Steroid
C27 20R 5B Monoaromatic Steroid
C27 20R aaa
C27 20R Triaromatic Steroid
C27 20S 5a Monoaromatic Steroid
C27 20S 5B Diamonoaromatic Steroid
C27 20S 5B Monoaromatic Steroid
C27 20S Ba Dia
C27STER_F
C28 20R 5a Monoaromatic steroid
C28 20R 5B Monoaromatic Steroid
C28 20R aaa
C28 20R Triaromatic Steroid
C28 20S 5a Monoaromatic Steroid
C28 20S 5B Monoaromatic Steroid
C28 20S Triaromatic Steroid
C28 22R Tricyclic
C28 22S Tricyclic
C28STER F
C29 17B21a Normoretane
C29 18a Neonorhopane
C29 20R 5a Monoaromatic Steroid
C29 20R 5B Monoaromatic Steroid

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C29 20R aaa
c29 20R aBB
C29 20S 5a Monoaromatic Steroid
C29 20S 5B Monoaromatic Steroid
C29 20S aaa
C29 22R Tricyclic
C29 22S Tricyclic
C29 Norhopane
C29BBAA_F
C29SR_F
C29STER_F
C30 17B21a Moretane
C30 17B21B Hopane
C30 20R aaa
C30 22R Tricyclic
C30 22S Tricyclic
C30 Diahopane
C30 Gammacerane
C30 Hopane
C30 Hopene (II)
C30 Oleanane
C31 22R 17B21B Hopane
C31 22R Hopane
C31 22R Tricyclic
C31 22S Hopane
C31 22S Tricyclic
C31HSR_F
C31RH_R
C32 22R 17B21B Hopane
C32 22R Hopane
C32 22S Hopane
C32HSR_F
C33 22R 17B21B Hopane
C33 22R Hopane
C33 22S Hopane
C34 22R 17B21B Hopane
C34 22R Hopane
C34 22S Hopane
C35 22R Hopane
C35 22S Hopane
C35C34_F
C35C34_R
DIAREG_R
GAC31R_R
GAMHOP_R
Homopregnane
MORHOP_R
NEONOR_R

NORHOP_R
OLHOP_R
PENT_F
PREGC27_R
Pregnane
S1S6_R
STER_F
STERPENT_F
TETC23_R
TRICY_F
TRIHOP_R
TRIMONO_F
TRIOCR_F
TRIOCR1_F
TRIOCR2_R
TSTM_R
TTM_F
XH_R
Isotope_C
C13 Whole Aromatic
C13 Whole Asphaltene
C13 Whole Kerogen
C13 Whole Liquid Organic
C13 Whole Oil
C13 Whole Resin
C13 Whole Saturate
C13 Whole Solid Organic
Isotope_C_Gas
C13 Carbon Dioxide
C13 Ethane
C13 iso-Butane
C13 iso-Pentane
C13 Methane
C13 n-Butane
C13 n-Pentane
C13 Propane
Rock-Eval
HI
OI
PC
PI
S1
S2

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S2S3
S3
SAMPWT
TMAX
TOC
Sox_Extract
CTYPE
EOM
EXSOL
EXTECH
MGAROM
MGASPH
MGBIT
MGCOL
MGNSO
MGSAT
PCTREC
RKWT
SATARO
VitRefl
RMEAN
RMODE
ROS

Appendix C. Login Procedure

Energy Lab Login Procedure

- Be sure sample names are clearly identified on samples before beginning.
- The login form is a Microsoft Excel spreadsheet with macros that run drop-down lists.
- Fill the login form out with required information: submitter information, analysis requested (must be in the English language), sample name (Client Field ID), Sample Type, Lat/Long Qualifier, latitude, longitude, elevation, depth (if applicable), and units. All other information such as Formation, Well Name, API number should be filled in if available but will not affect login.
- Items with a drop-down list must be selected from the list.
- The naming convention for file should be last name, underscore, and a unique identifier. (Example: mccord_Fl45).
- Send the login form to energylabs@usgs.gov
- Label the samples via 8 x 11 piece of paper in sample shipping container
- Send samples to:

  USGS/ Energy Login / Kelly Conrad
  Denver Federal Center
  Box 25046, MS 973
  Denver, CO 80225

Or:

- If you are located on the DFC you can bring samples to Building 20, Room G1111 login or outside Room A1115 login area. Put in proper location designated for sample submittal.
- Once samples arrive they will be matched up and checked against the login form for accuracy. If there are any discrepancies or questions you will be contacted by Login personnel before the samples are logged in.
- Samples will be submitted to the LIMS Manager and logged into the LIMS
- Confirmation of login will be sent to the submitter via email.
- The submitter must verify that all information is correct. If it is not, send email to energylabs@usgs.gov with a detailed description of what is wrong.

Note: All sample login and lab related questions should be sent to energylabs@usgs.gov

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Appendix D. Backup for LIMS

Backup Plan for Sample Master (LIMS)

Summary: Full backups of the following database files on server IGSKAECGAS007\SMV8:

System Databases
- master
- model
- msdb

SMV8 Databases
- SMV8
- SMV8Archive
- SMV8Temp

1. **Schedule**
   a. Full backup of databases – daily at 9:30 pm
   b. Backup files copied to T: Drive - daily at 11:00 pm
   c. Copy of backup files are deleted from the server after 20 days
   d. The two most current days backups are saved on T: drive for tape backup

2. **Backup Process –** A scheduled task is configured on IGSAKECGAS007 to run the SQLBackup.bat file. The SQLBackup.bat file runs the SMV8FullBackup SQL file. Each listed database file is backed up to \\IGSKAECGAS007\E:\SMV8_Backups
   a. Scheduled Task – "Backup SMV8 DB – Full" is scheduled to run at 9:30 pm everyday.
   b. "Backup SMV8 DB – Full" runs the SQLBackup.bat batch file which runs the SQL script, SMV8FullBackup.
   c. SMV8FullBackup script creates a full backup of each database and saves the backup to \\IGSKAECGAS007\E:\SMV8_Backups directory.
   d. All batch files and scripts can be found in the E:\Scripts directory on IGSKAECGAS007. And backup copies can be found in the T:\sample_master\smv8\backup\Scripts directory.

3. **Copy Files to T: Drive Procedure -** A scheduled task is configured on IGSAKECGAS007 to run the CopyBackupFiles.bat file. The CopyBackupFiles.bat file copies the most current files from \\IGSKAECGAS007\E:\SMV8_Backups to the T:\sample_master\smv8\backup directory.
   a. Scheduled Task – "Copy Backup Files to T Drive" is scheduled to run at 11:00 pm daily.
   b. "Copy Backup Files to T Drive" runs the CopyBackupfiles.bat batch file, which copies the most current backup files from E:\SMV8_Backups to T:\sample_master\smv8\backup directory.
4. **List of Batch Files and Scripts**
   a. **Backups:** "Backup SMV8 DB – Full" task -> "SQLBackup.bat" -> "SMV8FullBackup.sql"
   b. **Copy Files:** "Copy Backup Files to T Drive" task -> "CopyBackupFiles.bat"

Appendix E. Glossary

acceptance criteria — specified limits placed on characteristics of an item, process, or service defined in requirement documents

accreditation — the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one

accuracy — the degree of agreement of a measured value with the true or expected value of the quantity of concern (Taylor, 1987); see "bias"

analyte — a substance being determined in an analysis

ASTM Type I reagent water — Type I grade of reagent water is prepared by distillation or other equal process, followed by polishing with a mixed bed of ion exchange materials and a 0.2-mm membrane filter. It should have a minimum resistance of 18.0 MW-cm at 25 ºC (American Society for Testing and Materials, 2001, p. 107).

attachment — a secondary document that is attached to and supports the procedures presented in the standard operating procedure (SOP). An attachment provides supplementary information that is not included within the body of the SOP but is necessary to correctly perform the procedure or clarify statements made within the SOP. Attachments must be referenced within the text of the SOP. Attachments go through the same review procedures as the rest of the SOP and are part of the SOP. Examples of attachments are manifold diagrams, computer instructions, acceptance ranges for specific quality-control samples, forms required to complete a procedure, and formatting instructions.

audit — a systematic evaluation to determine the conformance to quantitative specifications of some operational function or activity

backlog— all samples in the laboratory that are recognized, verified, and tracked in the laboratory information management system (LIMS)where analysis are incomplete.

bias — systematic error inherent in a method or measurement system. The error can be positive [for example, contamination or spectral interference] or negative [for example, analyte loss or signal suppression] It differs from random error, which shows no such consistent or systematic deviation.

blank — a sample that contains no measurable amount of analyte , and is used to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

blind sample — a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process

calibrate — to check, adjust, or determine by comparison to a standard

calibration — the set of operations that establishes, under specified conditions, the relation between values or quantities indicated by a measuring instrument or system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. In calibration of support equipment, the values realized by standards are established through the use of reference standards traceable to the International System of Units.

calibration curve — the graphic relation between the known values, such as concentrations, of a series of calibration standards and their analytical response

calibration standard — a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration

certified reference material (CRM) — a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body

continuing calibration verification standard (CCV) — a standard solution used in instrumental analysis to check instrument stability in relation to the calibration standard curve. Prepared from the same materials in the same manner as the calibration standards. Concentration of the CCV should be chosen to allow easy review by the analyst and is typically in the midrange of the calibration curve.

control limit — statistically derived values used to get acceptable ranges for quality-control samples analyzed in conjunction with environmental samples

corrective action — action taken to eliminate the cause(s) of an existing nonconformity, defect, or other undesirable situation to prevent recurrence

data — information from a measurement or observation that may be used for calculating, reasoning, or discussion. Data may be in the form of numbers, words, images, or other forms.

data integrity — the ability to maintain and/or preserve a piece of data or information, and to recreate a piece of data or information should accidental loss occur

data review — the process of validating and/or approving data after analysis or processing. Once approved the data are flagged as ready to be released to the customer.

double-blind sample — a sample submitted to evaluate performance with concentration and identity unknown to the analyst

F-pseudosigma (Fs) — a nonparametric, resistant measure of data spread defined as the interquartile range of the data divided by 1.349 .Fs and the standard deviation of the data will be nearly equivalent if the data have a near-normal distribution.

good automated laboratory practices (GALP) — principles and practices to ensure data integrity in automated laboratory operations

good laboratory practices (GLP) — general guidelines or formal regulations for performing basic laboratory operations or activities that are known or believed to influence the quality and integrity of the results

hazardous samples — samples considered to contain high concentrations of contaminants and may have deleterious effects on human health or the environment

hazardous waste — a solid, liquid, or contained gaseous material that is no longer used or that no longer serves the purpose for which it was produced and could pose dangers to human health and the environment after it is discarded (Colorado Department of Public Health and Environment, 1995)

holding time (maximum allowable holding time) — the maximum time(s) that a sample may be held prior to analysis and its analytical results still be considered valid

instrument detection limit (IDL) — the concentration equivalent to a signal from an analyte of interest, which is the smallest signal that a particular instrument can distinguish from background noise. The IDL may be used for statistical data analysis and comparing the attributes of different instruments. It is determined on samples that have not gone through any sample preparation steps.

internal standard — a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the bias and variability of the applied analytical test method.

laboratory control sample (LCS)— a blank spiked with verified known amounts of analytes from a source independent of the calibration standards, or a sample containing known and verified amounts of analytes. It is generally used to establish intralaboratory or analyst-specific bias and variability or to assess the performance of all or a part of the measurement system.

laboratory information management system (LIMS) — a centralized data management and storage system that maintains data security, and data and information integrity. The system gathers or receives data from other analytical systems, monitors quality-control measures, records operating procedures, and processes and diseminates information and data of known quality.

laboratory reporting level (LRL) — equal to twice or more the annually determined long-term method detection level (LT–MDL). The LRL controls false negative error. The probability of falsely reporting a nondetection for a sample that contained an analyte at a concentration equal to or

greater than the LRL is predicted to be no more than 1 percent. The LRL concentration will be reported with a “less than” (<) remark code for samples in which the analyte was not detected.

long-term method detection level (LT–MDL) — a detection level derived by determining the standard deviation (or F-pseudosigma) of a minimum of 24 method detection limit (MDL) spike-sample measurements over an extended period. LT–MDL data are collected continually to assess year-to-year variations in the LT–MDL. The LT–MDL controls false positive error.

marginal exceedance (ME) — laboratory control sample (LCS) data that are beyond the LCS control limit of three standard deviations around the mean, but within the ME limits of three and four standard deviations around the mean. If a large number of analytes is in the LCS, it is statistically likely that a few will be outside control limits. This may not indicate that the system is out of control and corrective action may not be necessary. Upper and lower ME limits can be established to determine when corrective action is necessary.

matrix — the component or substrate that contains the analyte of interest; the substrate of a sample.

method blank — a clean sample (matrix similar to the batch of associated samples and is free of the analyte of interest) processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

method detection limit (MDL) — the minimum concentration of a substance (an analyte) that can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

minimum reporting level (MRL) — smallest measured concentration of a constituent that may be reliably reported by using a given analytical method . It is the "less than" value reported when an analyte either is not detected or is detected at a concentration less than the MRL

National Environmental Laboratory Accreditation Conference (NELAC) — a cooperative association of states and Federal agencies, formed to establish and promote acceptable performance standards to operate environmental laboratories. The standards cover analytical testing of environmental samples and the laboratory accreditation process

National Environmental Laboratory Accreditation Program (NELAP) — the National Environmental Laboratory Accreditation Program (NELAP) implements the standards developed by the National Environmental Laboratory Accreditation Committee (NELAC). States and Federal agencies serve as accrediting authorities, with coordination facilitated by the U.S. Environmental Protection Agency to ensure uniformity. Accreditation by one NELAP accrediting authority is mutually recognized by other State and Federal accrediting authorities approved under NELAP.

negative control — measures taken to ensure that a method, its components, or the environment do not cause undesired effects, or produce incorrect results from possible contamination during preparation, processing, and instrumental analysis. For example, a method blank is a negative control.

performance audit — the routine comparison of independently obtained quantitative measurement system data with routinely obtained data to evaluate the proficiency of an analyst or laboratory

performance evaluation (PE) — proficiency testing by evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source and determining a laboratory's calibration or testing performance by means of interlaboratory comparisons. The results of PE samples show whether or not a laboratory has an analytical bias, if the bias is continuing, and/or if the bias is increasing. If results are not within the control limits, a sample is analyzed again.Test samples are prepared by spiking known concentrations of select analytes into a well-characterized matrix. Typically, PE samples are made in a single matrix such as an aqueous, solid, or oil matrix. PE samples can be distributed as single- or double-blind samples. PE samples are used to determine a laboratory's accuracy as it relates to the execution of a particular analytical method.A PE program provides controlled and standardized environmental samples to participating laboratories for analysis,

reporting of results, statistical evaluation of the results in comparison to peer laboratories, and the collective demographics and results summary of all participating laboratories.

pH — the logarithm of the reciprocal of the hydrogen-ion concentration in gram atoms per liter. It is numerically equal to 7 for neutral solutions and increases with increasing alkalinity and decreasing with increasing acidity. The pH scale ranges from 0 (acidic) to 14 (basic). Each unit of change represents a ten-fold change in hydrogen ion concentration.

positive control — measures taken to ensure that a method and/or its components are working properly and producing correct or expected results. A laboratory control sample is typically used as a positive control.

precision — the degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions or a measure of the degree of agreement among replicate analyses of a sample, usually expressed as the standard deviation.

preservation — refrigeration and/or reagents added at the time of sample collection or later to maintain the chemical and/or biological integrity of the sample

procedure — a process, method, or instrument analysis.

protocol — a detailed written procedure for a field and/or laboratory operation (for example, sampling, analysis) that must be strictly followed.

quality assurance (QA) — a set of operating principles that, if strictly followed during sample collection and analysis, will produce data of known and defensible quality. That is, the accuracy of the analytical result can be stated with a high level of confidence. It is a definitive program for laboratory operation that specifies the requirements that will produce data of known bias and variability. Included in QA are quality control and quality assessment. In a more general context, QA is an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

quality control (QC) — a set of measures within a sample analysis methodology to assure that the process is in control the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users

range — the difference between the minimum and the maximum of a set of values

raw data — any original factual information or unprocessed data from an analysis, measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, photography, microfilm, microfiche, computer printouts, magnetic media, dictated observations, and recorded data from automated instruments.

reagent blank (method reagent blank) — a sample consisting of reagent(s), without the specified analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps to any interference or contamination.

reference material — a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

reference standard — a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived

replicate analyses (Duplicate) — the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time

sample — one or more portions of a liquid, gas, or solid material (including biological tissues) taken for a specific purpose from a batch, lot, process stream, or from a natural geologic setting in order to determine the chemical, physical, mechanical, or other quality characteristics of the material, or combination thereof.

sample preparation — any procedure or process performed on a sample before analysis. This may be done by the client at the time of sample collection or employees at the USGS Laboratory after login of samples, or both.

sensitivity — the capability of a method or instrument to discriminate between measurement responses representing different levels (for example, concentrations) of an analyte of interest.

spike — a known mass of specified analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality-control purposes

standard operating procedure (SOP) — a document that describes the analytical methods to be used in the laboratory in sufficient detail that a competent analyst unfamiliar with the method can conduct a reliable review and/or obtain acceptable results.Where applicable, an SOP may include title of reference, consensus analytical method; sample matrix or matrixes; method detection level (MDL); scope and application; summary of SOP; definitions; interferences; safety considerations; waste management; apparatus, equipment, and supplies; reagents and standards; sample collection, preservation, shipment, and storage requirements; specific quality control practices, frequency, acceptance criteria, and required corrective action if acceptance criteria are not met; calibration and standardization; details on the actual test procedure, including sample preparation; calculations; qualifications and performance requirements for analysts (including number and type of analyses); data assessment/data management; references; and any tables, flowcharts, and validation or method performance data

standard reference material (SRM) — certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization, and characterized for absolute content, independent of analytical test method

standard reference sample (SRS) — a sample that is prepared to check and monitor inorganic or organic analytical systems.

surrogate — a substance with properties that mimic the analyte(s) of interest. It is unlikely to be found in environmental samples and is added to them for quality-control purposes.

test — a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure

traceability — the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons

variability — random error in independent measurements as the result of repeated application of the process under specific conditions. Variability can be statistically described by the standard deviation. All data contain some experimental error and individual measurements change or fluctuate within limits. Precision is a measure of variability in experimental data.

verification — confirmation by examination and provision of evidence that specified requirements have been met

waste — a solid, liquid, or contained gaseous material that is no longer used or no longer serves the purpose for which it was produced.

APPENDIX 3. Report of the USGS NWQL audit of ERP Denver labs.

Audit Report
USGS Geologic Discipline Energy Resource Program Geochemistry Laboratory
April 27 – 29, 2009

NWQL Audit Number: CLAB 2009.14
Audit Team Members: Lisa Bressler and Jeanne Hatcher, NWQL Quality Assurance Section
Participating EGL Personnel:
Jamey McCord
William Betterton
Lisa Brazeau
Khanh Doan
Steve Groves
Tammy Hannah
Toni Kennedy
Paul Lillis
Mark Pawlewicz
Augusta Warden

Background:

At the request of USGS Geologic Discipline Energy Resource Program Geochemistry Laboratory (EGL), personnel from the National Water Quality Laboratory (NWQL) Quality Assurance Section (QAS) performed an audit of EGL to evaluate the effectiveness of the EGL quality system and the implementation of the Quality Control Manual (QCM). The scope of the audit included the following procedures and documents:

EGL Quality Control Manual version 1, approved and implemented April 24, 2009
Various analytical standard operating procedures

The audit report is a documentation of the auditor's observations, suggestions, and concerns for EGLs' use. No response is required.

Summary of audit:

NWQL personnel conducted an audit of EGL on April 27 – 29, 2009. The audit began with an opening meeting attended by USGS and EGL personnel. In attendance from NWQL were Doug Stevenson, Lisa Bressler and Jeanne Hatcher; in attendance from EGL were Jamey McCord, Chris Potter, Margaret Ellis, Ron Affolter, Steve Groves, and Paul Lillis. The details of the audit, including scope and schedule were discussed and were followed by a laboratory tour.

The audit was conducted with a preliminary review of the QCM and various SOPs. On site activities included laboratory walk through, interview of laboratory personnel and

review of additional support documentation such as logbooks, training records, and sample tracking systems.

A debriefing meeting was held at the end of the audit to outline the concerns and strengths. In attendance from NWQL were Doug Stevenson, Lisa Bressler and Jeanne Hatcher. Attending from EGL were Jamey McCord, Chris Potter, Margaret Ellis, Paul Lillis and Steve Groves.

Major Concerns

1. EGL management should support implementation of the QCM in the laboratory.

2. In the QCM Section 1, Laboratory Management, there are several positions that are presently performed by one (1) person. The role of the QA/QC Manager needs to be independent of other positions, so as not to provide a conflict of interest.

3. A sample backlog presently exists. We recommend that turnaround times be identified and implemented to reduce and maintain the reduction of sample backlogs.

4. Compliance with the QCM should be promoted. For example, the QCM identifies requirements for laboratory notebooks that need to be implemented labwide.

5. Previous internal annual in-house audits have identified findings that have not yet been addressed. Corrective actions for these findings need to be identified and implemented.

Documentation Issues

1. The SOP or the QCM should include how often and the means to verify calibration of balances. Most analysts verify the calibration by performing the balances internal self test and occasionally using weights. The balances are calibrated annually utilizing an outside source. It is suggested that it should be defined as to the frequency of checks, how checks are performed, and that the documentation of the checks be maintained in a table in a balance logbook.

2. There were many instances of unlabeled or mislabeled bottles/containers, including several containers which had expiration dates from years ago. The labeling of solvents, reagents, reference materials, and standard containers should be consistent throughout the laboratory and in accordance with the QCM.

3. The QCM clearly states defined expectations of what is to be documented in standards, maintenance, and analytical system log books; however these requirements need to be put into practice.

General

1. Having a common sense attitude towards safety assumes everyone has common sense. Experience shows that not everyone is thinking the same way. SOPs should include clear direction regarding when and what type of PPE must be worn. Generally, accepted laboratory practice discourages eating and drinking in the laboratory.

2. SOPs should have footer information that contains at a minimum the version/revision, date of revision, and page X of Y. This was completed prior to the end of the audit.

3 Include a glossary in the QCM to define terms and acronyms. This was incorporated into the QCM prior to the end of the audit.

4. Many balances have out of date annual calibration verification stickers.

5. SOP revisions are passed on to analysts and sample preparation staff verbally and the changes are not always incorporated into the approved SOP. This is a document control concern.

6. The auditors reviewed the QCM with the QA/QC manager and all corrections, changes, and suggestions were incorporated prior to the end of the audit.

7. The entire laboratory would benefit with a complete housecleaning of all solutions, reagents, standards, and reference materials. If items cannot be verified with respect to their quality, expiration, and traceability, then they should be disposed of appropriately.

8. It appears that there is a lot of excess equipment. Some of it is infrequently used and some is not used at all. Equipment needs should be evaluated to determine if maintaining unused equipment meets the requirements of the mission of the EGL.

Strengths

1. All of the analysts that we spoke to were very knowledgeable regarding their instruments and methods, and there was open communication between them and the auditors.

2. The Sample Master Laboratory Information and Management System (LIMS) is an enormous asset that facilitates all QA/QC and sample tracking. The following is a list of major strengths of the LIMS:

- Generation of control charts by analyst, instrument, date range or other information
- Automatic accuracy and precision calculations for MS, MSD, and LCS results
- Automatic limit checking on data entry
- Full audit trail from initial sample to results, including changes
- Ability to attach files from different formats

3. The Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and the Gas Chromatography-Mass Spectrometry (GC-MS) laboratories should be used as representations of how the implementation of QCM requirements should be implemented.

Conclusion

The quality system is still in its infancy and complete implementation will take a number of months. It is suggested that the following items be re-audited in a year to verify compliance with the QCM. If at that time, items are not put into practice, an audit report should be written, provided to the Service Center Director and responses should be documented and tracked in accordance with the QCM.

- Updated SOPs in appropriate laboratories
- In house audit findings are documented and responses tracked to closure
- Individual training files created and up to date
- Equipment, standards and balance logbooks meet QCM requirements
- Standards and reagents labeling meets QCM requirements

Overall, the audit verified that the full implementation of the QCM will provide EGL with sufficient documentation and traceability to support the quality of data produced. It is important that EGL management fully support the QCM implementation across the laboratory.

Appendix 4. Questionnaire Form (4A) and Summary of Responses (4B)

Appendix 4A.
Energy Resources Program Geochemical Laboratory Questionnaire
May 2009

In response to data quality concerns that came to light last year in our Geochemical Laboratory, the Energy Resources Program (ERP) is undertaking a thorough review of geochemical laboratory operations, services, and procedures, including quality assurance. The review is being coordinated by a team consisting of Chris Potter, Jamey McCord and Paul Lillis (Energy Resources Science Center in Denver); Jim Coleman, Terry Lerch and Sue Tewalt (Energy Resources Science Center in Reston); and Doug Duncan (ERP Associate Program Coordinator). A significant component of the review was a QA/QC audit of our Denver labs, performed by QA specialists for the USGS National Water Quality Lab. This audit was completed in early May, 2009, and the audit report is included as another email attachment. We urge you to read it.

As part of this process, the Lab Review Coordination Team is soliciting feedback from scientists in the two Energy Resources Science Centers, and this is the purpose of the following questionnaire. It is important for us to understand the analytical needs and concerns of the ERP scientists.
If you could take the time to reply to this short list of questions, it would be greatly appreciated. Thanks in advance. Please return the completed questionnaire to EnergyLabs@usgs.gov by June 12, 2009.

QUESTIONNAIRE BEGINS ON THE NEXT PAGE.

Abbreviations used in the questionnaire:
LOI - loss on ignition
ASH - ashing
AA - atomic absorption
ICP - inductively coupled plasma
ICPMS - inductively coupled plasma-mass spectrometry
XRD - x-ray diffraction
GCFID - gas chromatography flame ionization detector
GCMS - gas chromatography mass spectrometry

Name _____Date_____

(1) Looking forward (next 1-3 years), please estimate the total number of samples per year that you anticipate submitting, for each of these sample types.

Sample Type	Annual Number of Samples
Oil	
Gas	
Rock	
Coal and Ash	
Other (Filter, Biologic, etc.)	

(2) Here is a list of current analytical techniques for which we have standard operating procedures, divided into inorganic and organic analyses. Please comment below on whether or not you feel this is an appropriate suite of analyses to be performed routinely by ERP labs, and list any additional analyses you would like to see in the future (rare earths, for example?).

Inorganic	Organic
LOI750	API Gravity
ASH750	Elemental Analysis (C,H,N,O,S)
Moisture	Gas Composition
Hg	GCFID (Aromatics, Saturates)
Se	GCFID (Whole Oil)
ICP_MajorForSolids (Al_2O_3, CaO, Fe_2O_3, K_2O, MgO, Na_2O, P_2O_5, SiO_2, SO_3,TiO_2, Ba, Sr)	GCMS on Biomarkers (276 Possible)
ICPMSTrace for solids (As, Be,Bi, Cd, Co, Cr, Cs, Cu, Ga, Ge, Mn, Mo, Nb, Pb, Rb, Sb, Sc, Th, Tl, U, V, Y, Zn	Carbon Isotopes on solids and gas
Sulfur	Vitrinite Reflectance
XRD	

Your Comments:

(3) For those who have future geochemical analytical needs, please indicate below how likely you are to submit future samples:

to the Denver inorganic lab?

Likely _____ Not likely _____

or to the Denver organic lab?

Likely _____ Not likely _____

If “not likely,” please indicate why that is the case:

Thanks for your participation and feedback.

Appendix 4B.

Energy Resources Program Geochemical Laboratory Questionnaire May 2009: Summary of Responses.

Total number of responses: 25

The responses are summarized below using the questionnaire format.

Name: **Summary of USGS Energy Scientists' Responses**
Date: **May/June, 2009**

(1) Looking forward (next 1-3 years), please estimate the total number of samples per year that you anticipate submitting, for each of these sample types. *Numbers listed are total numbers reported.*

Sample Type	Annual Number of Samples
Oil	420
Gas	675
Rock	933
Coal and Ash	1165
Other (Filter, Biologic, etc.)	2650

(2) Here is a list of current analytical techniques for which we have standard operating procedures, divided into inorganic and organic analyses. Please comment below on whether or not you feel this is an appropriate suite of analyses to be performed routinely by ERP labs, and list any additional analyses you would like to see in the future (rare earths, for example?).
For list, see Appendix 4A.

Summary of Comments on the suite of analyses offered:

# of respondents who indicated that the suite of analyses is appropriate: 10

Other new analyses requested:

Multiple requests:

∂H deuterium for natural gas (6 requests)
REE in coal and ash (6 requests)
Cl in coal and ash (4 requests)
C1-5 for natural gas (4 requests)
∂13C (3 requests)
F, B, Ni for coal (2 requests)

Other requests:

C4-7 for natural gas
Quantitative XRD
GCMS biomarkers, quantitative (oil)

GS FID, quantitative (oil)
GC/FPD method for organic sulfur
Inorganic water composition
Sr
Se
Mn
Br, Li and Zr, REE, PGEs for coal
C for both coal and coal ash
Dissolved Organic Compounds (water)
H and O isotopes on produced water samples
Major anions (IC), major cations (ICP-AES), Total Radium, total Radon, Ra isotopes (226,228), Uranium isotopes (234,238, 235), Rn isotopes, S isotopes (34,32), gross alpha and beta, thorium isotopes (228, 230, 232), lead isotopes (210 and 214), Bi(214), oxygen isotopes, FTIR, Fe speciation, U speciation, As speciation, S speciation

(3) For those who have future geochemical analytical needs, please indicate below how likely you are to submit future samples:

to the Denver inorganic lab? *(numbers reflect responses from users or possible users of the inorganic lab)*

Likely -12 Not likely-2 Maybe-3

or to the Denver organic lab? *(numbers reflect responses from users or possible users of the organic lab)*

Likely -16 Not likely-2 Maybe-1

If "not likely," please indicate why that is the case:

The negative and noncommittal responses were accompanied by concerns about data quality, QA/QC documentation, turnaround time, lab cleanliness, LIMS accessibility.