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1. Manual overview

This document controls the implementation of policies and procedures utilized by the Energy Geochemistry Laboratory (EGL) to assure quality of data, emphasizing continuous commitment to improve both quality and efficiency as well as to identify problems and prevent their recurrence. The EGL recognizes that Quality Assurance (QA) is an integral part of routine laboratory analysis. Analytical results must have a clearly defined pedigree, allowing a knowledgeable person to determine who performed the analytical work, when it was performed, how it was performed, and to track instrumental data output to final analytical result.

The EGL Quality Assurance Manual provides basic requirements that are intended to conform to the policy in the USGS Fundamental Science Practices. Although many aspects of the EGL QA Manual are similar to other organizations' QA practices, the QA Manual is not meant to satisfy the requirements of any outside organization or accreditation agency.

In addition to the requirements and policies specified, USGS employees are required to comply with the following related policies:

Industrial Hygiene - Laboratory Protection Program (USGS Manual 445-2-H Chapter 21)

USGS Code of Scientific Conduct (USGS Manual 500.25 Part 7)

2. Requirements

The primary requirements that guide the development of this manual stem from the USGS Manual Section 502.2 - Fundamental Science Practices: Planning and Conducting Data Collection and Research. The subsections that are applicable to laboratory quality assurance are:

Part 4. Policy

A. Data collection and research activities are carried out in a consistent, objective, and replicable manner that has been vetted through a vigorous and open process of peer review to ensure that the best possible results are achieved and that there are no weaknesses or errors in data or conclusions.

This requirement is met by Section 6 – Methods in this document.

B. Standard USGS methods are employed for distinct research activities that are conducted on a frequent or ongoing basis and for types of data that are produced in large quantity. Methods must be documented to describe the performance of the method and the quality assurance procedures applied. When scientific reason justifies the use of alternative or experimental methods, such methods are documented and the rationale for their use is clearly stated.

This requirement is met by Section 6 - Methods and Section 10 – Records in this document.

C. Methods and techniques used to conduct data collection and research activities are published in information products that are easily accessed and are available in a manner that enhances the scientific reputation of the Bureau and best serves the whole public....

This requirement is met by Section 6 – Methods in this document.

D. Data collected by the USGS and techniques used by USGS scientists should utilize or reference national and international standards and protocols where they exist and when they are relevant and appropriate. For datasets of a given type, and where national or international metadata standards exist, these data are indexed with metadata that facilitates access and integration.

This requirement is met by Section 6 - Methods and Section 10 – Records in this document.

In addition to the Fundamental Science Practices cited above, USGS Manual 432-1-55: Geology Discipline Research Records Schedule, section 1900-01a Significant Research Records, defines records as:

Records include original observations such as ... analyses and observations made with electronic or other equipment, laboratory notebooks, databases that contain scientific observations ... and any other research related documentation.

The requirements to preserve these records are met by Section 10 – Records in this document.

3. Organization and Responsibilities

Quality assurance depends in part on clearly defined organization and personnel responsibilities. In order to function smoothly and efficiently, each role in the organization must be defined and the personnel assigned to fulfill those duties must be informed as to their responsibilities. The functional organization chart for the EGL is shown in Figure 1. Note that there is not necessarily a one-to-one correspondence between the boxes on the chart and specific personnel. In other words, a single person may perform more than one function.

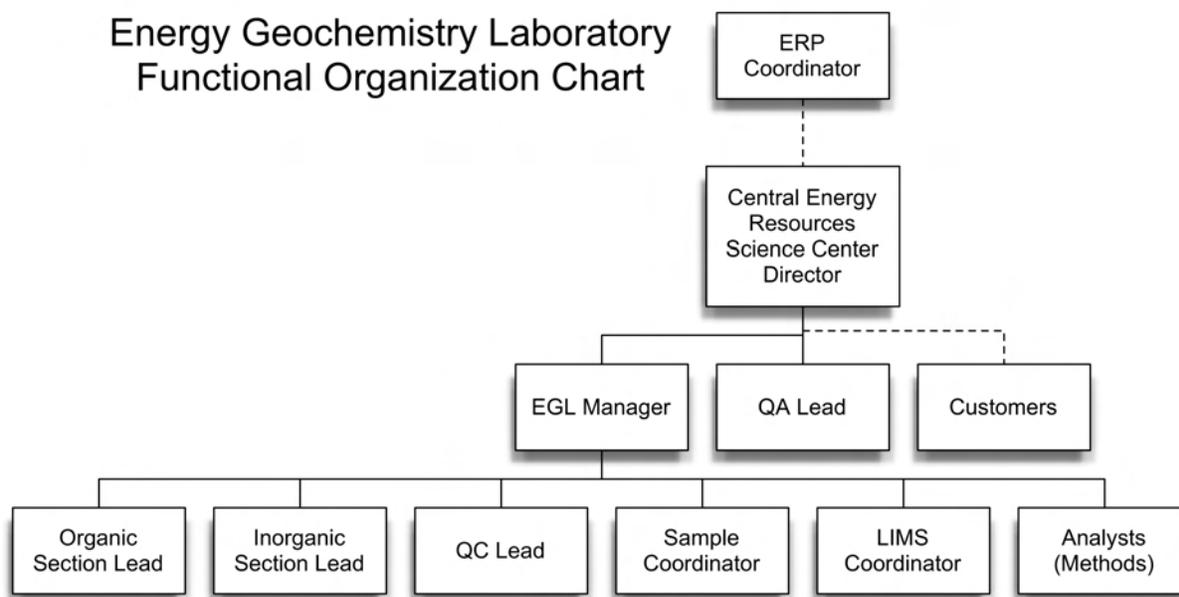


Figure 1. EGL Functional Organization Chart. ERP – Energy Resources Program; QC – Quality Control; LIMS – Laboratory Information Management System.

The responsibilities of each function are listed below.

Energy Resources Program Coordinator

- Coordinates budget and personnel decisions with Energy Resources Science Center staff
- Arranges for audits of EGL QA program
- Facilitates communication of issues or complaints with the EGL Manager

Central Energy Resources Science Center Director

- Manages the integration of the EGL with the Central Energy Resources Science Center and the Energy Resources Program.
- Coordinates budget and personnel decisions with the Energy Resources Program.
- Ensures that standards for scientific quality are met; that is, methodology is documented, and accepted metadata standards are used (USGS Circular 1367).

EGL Manager

- Defines laboratory operations consistent with analytical needs and budget constraints.
- Assigns personnel to functions as dictated by analytical needs.
- Ensures that Analysts are properly trained.
- Reports on laboratory operations to management on a regular basis.
- Prepares budgets for all EGL operations.
- Facilitates purchases of major equipment.
- Checks manuscripts that publish EGL data for consistency with LIMS and changes data status in LIMS to public release (if identified otherwise).

QA Lead

- Designs a QA program consistent with USGS policy and works with staff to implement the program.
- Maintains a QA Manual (this document) describing the QA program.
- Receives notifications of suspect data that are quarantined, determines course of action such as re-analysis, and issues concerns if a condition is identified.
- Tracks issues and corrective actions.
- Reviews Methods.

QC Lead

- Uploads formatted data into the Laboratory Information Management System (LIMS), submitted by Analysts or from outside labs.
- Coordinates the analysis of Quality Control samples with Analysts and the LIMS Coordinator.
- Approves release of data to customers based on Analyst validation and analysis of QC samples identified in Method documents. Any data that are determined to be suspect and therefore not to be released shall be quarantined and notification of the condition shall be made to the QA Lead.
- Reports on Quality Control results on a regular basis.
- Tracks EGL participation in proficiency testing programs.

- Submits "blind" samples in order to test methods that identify QC standards and compiles results of this "blind" testing. Standards to be submitted as "blinds" should either be those QC standards described in the Method document or other samples determined to be suitable for use as standards based on consultation with the Analyst that performs the Method.
- Responds to data issues, requests re-analysis, and performs updates to the LIMS, ensuring that tracking of older versions is maintained.

Organic Section Lead

- Requests budget for operational expenses incurred by Organic Section.
- Facilitates repairs, maintenance, and purchase of consumables for Organic Section instrumentation.
- Advises Analysts as necessary of current backlog of analytical work.

Inorganic Section Lead

- Requests budget for operational expenses incurred by Inorganic Section.
- Facilitates repairs, maintenance, and purchase of consumables for Inorganic Section instrumentation.
- Advises Analysts as necessary of current backlog of analytical work.

Sample Coordinator

- Receives samples that are submitted to the EGL.
- Facilitates communication between customers and the LIMS Coordinator regarding sample receipt, login, and requests for analytical work.
- Determines if samples are properly packaged and identified, and if additional physical preparation is necessary.

LIMS Coordinator

- Requests budget for operational expenses incurred by LIMS operations.
- Logs samples into the LIMS in coordination with the Sample Coordinator, and Inorganic and Organic Section Leads.
- Verifies that requested analytical work is available and coordinates the requests with the Section Lead.
- Confirms successful login with the customer and with appropriate Section Lead(s).

- Creates a backlog report on a regular basis.
- Responds to requests for data from customers and external organizations.
- Maintains LIMS and communicates with the LIMS supplier on issues or updates.
- Designs routines to import data from outside sources as necessary in order to ensure that the LIMS is a comprehensive data source for all Energy Resources Program scientific staff.

Analysts

- Perform physical or chemical sample treatments per established Methods.
- Perform instrumental analysis according to established Methods.
- Ensure that instrumental (raw) data are protected from accidental erasure or inadvertent loss.
- Prepare data in the appropriate format for upload to the LIMS.
- Validate data entered into the LIMS for the instrumental analysis.
- Maintain instrumentation in good working order, communicating any off-normal conditions or needed repairs to the appropriate Section Lead.
- Maintain laboratory notebooks for all instrumentation that is used in the preparation of analytical data submitted to the LIMS.
- Write Method documentation and handle changes to Methods as necessary
- Document training received on EGL Record of Training (Attachment 1)

Customers

- Provide appropriate metadata for samples submitted on required form.
- Request analytical work.
- Consult with Lab Manager regarding analytical needs.
- Notify Sample Coordinator of any hazardous or unusual sample characteristics or matrices, and of unusual analyte concentrations, if known.
- Notify QA Lead of concerns or issues with data.
- Are responsible for proper packaging, labeling, and shipment of samples.

- Review analytical results within reasonable timeframe.
- Notify Sample Coordinator of desired sample disposition.
- May submit their own QC samples as part of a job.
- Identify manuscripts containing EGL data on publication routing sheets (or via mechanism acceptable to customer's Science Center) in order to facilitate a final check of data correctness prior to public release

Regarding this last bullet, customers and principal investigators are ultimately responsible for the integrity of research data. Although this QA Manual ensures that the EGL applies the proper procedures to provide the highest quality analytical data, ultimate responsibility for design of an investigation and requesting the appropriate analyses to answer a research question resides with the individual research customer. For this reason, customers are encouraged to discuss research activities directly with EGL staff, especially in cases involving a new type of study or unique sample matrices.

4. Document Control

Controlled documents include:

- QA Manual (this document)
- EGL Method documents

These documents shall have embedded revision histories and strict version control. Methods, including version, are irrevocably linked to data in the LIMS.

5. Training

Training is expected to be an important component of the EGL, with staff requesting training to improve efficiency and utilization of instrumentation as well as knowledge of new methods, new software, and new instrumentation.

Demonstration of Proficiency

A demonstration of proficiency documents the Analyst proficiency in each Method that they perform. Proficiency of the Analysts in assigned Methods is maintained by: (1) ensuring that each Analyst has read, understood, and is using the latest version of the applicable Method, and (2) a check of proficiency by the Analyst in assigned Method using QC standards specified in the Method, preferably submitted "blind". Each of these items shall be documented on the EGL Record of Training Form (Attachment 1). Analysts currently working to Methods or SOPs already in place on the effective date of this document are not required to demonstrate proficiency in these Methods or SOPs.

Ethics

Training requirements for ethics are the same as for any USGS employee. Refer to USGS Code of Scientific Conduct (USGS Manual 500.25 Part 7).

Safety

Training requirements for safety are the same as for any USGS employee that works in a laboratory environment. Refer to Industrial Hygiene - Laboratory Protection Program (USGS Manual 445-2-H Chapter 21).

All training that is relevant to this QA Manual, including reading assignments, shall be documented by personnel receiving training on the EGL Record of Training Form (Attachment 1).

6. Methods

Methods are the fundamental documents that describe the procedures utilized by the EGL to acquire chemical data and (or) perform sample preparation. Methods are written for all routine analytical procedures and typical sample preparation. The individual Methods, including version, are associated with data that are generated using these Methods in the LIMS. Therefore, deviations from established Methods must be documented by the Analyst in data files and communicated to the LIMS Coordinator. In general, if an established Method requires frequent deviations, the applicable Method should be modified to accommodate this variability.

Method documents are controlled documents with appropriate versioning and revision history. Methods may include attachments, such as instrument documentation. Method documents are to be published on the EGL web site; however, it is expected that attachments to Methods may be kept off-line due to copyright restrictions or size.

Method documents require a specific review and approval process to ensure adequate review and appropriate approvals in order to be published. Attachment 2, EGL Method Review and Approval, controls this process and documents the review.

Methods or procedures in effect as of the date of this QA Manual revision shall remain in effect without modification. Methods currently in use shall be modified to comply with the provisions of this QA Manual within one year of the effective date of the QA Manual.

Method Outline

The headings and general format of EGL Methods should be consistent in order that all scientific staff can easily find information related to EGL procedures. The annotated outline for EGL methods is presented here.

1. Introduction

This section should begin with a brief statement of why analyses or procedures are performed and what they are useful for. If the Method follows a method published elsewhere (ASTM, EPA, USGS), make that statement and reference the source method. If a Method is stated as being substantially the same as a published method, then the significant differences, if any, should be described here.

2. Interfaces with Other Methods

This section documents any prerequisite Methods. If this Method relies on products or results generated by another Method, then make that statement and reference the other Method. Examples include chemical separation or preparation Methods that are performed prior to this analysis, and weighing of samples. All Methods will likely interface with the Method for sample control, labeling, and disposition.

3. Materials and Equipment

This section will normally contain a list of equipment and chemicals that are used in performing the Method. Note that standard laboratory containers do not need to be listed unless their specific characteristics impact the analysis.

4. Procedure

This section should describe, at a level suitable to another trained Analyst, the typical steps that are followed to perform an analysis or batch of analyses, or describe a preparation or sample treatment. Enough detail should be included that another Analyst could perform the analysis. However, steps that are only relevant to a specific model of instrument should probably be moved to an attachment. Instrument manuals, or portions thereof, can be used, but must be as an attachment (these attachments will not be included in published Methods in order to comply with copyright restrictions).

5. Calibration and Quality Control Samples

If the Method utilizes instrumentation, this section should describe how it is calibrated, what standards are used, and what determines the need for re-calibration. QC samples or check standards should be documented here, including their sources and references for their values.

6. Limits, Precautions, and Interferences

This section should describe any limits or known interferences which could affect the data; also, any special precautions should be discussed here. Limits are typically restrictions on sample matrices or range restrictions on analyte values such as detection limits. Precautions are either actions to be avoided to protect the integrity of samples or data, or actions or warnings to prevent bodily harm that are specific to the Method.

7. Acceptance of Data

Specify criteria that will be used to determine if data generated by this method are acceptable, such as QC checks, duplicates, etc. Also, specify the expected precision of the analytical results

8. Data Handling and Transfer

In this section, discuss the detailed handling of data. Specifically, the steps required to get the data from the instrument to the LIMS. If data are transcribed by hand, describe what checks are utilized to ensure accurate transcription. If calculations are performed off-line (outside of the instrument software system), specify them completely. This section should document all steps involved in getting from raw data generated by an instrument to final values as reported in the LIMS.

9. References

List any references cited.

10. Attachments

List any attachments (or state that there are none). These could include detailed lists of analytes or instrument-specific operating instructions.

11. History of Changes

If this is the first version of the Method, this section should read “Revision 0: initial issue.” Otherwise, this section should be a list of changes made with each revision.

12. Authorship and Approvals

This page contains signatures of approving personnel. Required approvals are author(s), reviewer(s), QC Lead, Lab Manager, and Science Center Director. Attachment 2 shall be utilized for this page of the Method.

7. Instrumentation

Instrumentation is key to obtaining analytical chemical data. Detailed operation of individual instrumentation is provided in the various Method documents. There are certain requirements, however, that are relevant to all instrumentation in the EGL.

Logbooks

A logbook is maintained for each instrument and analytical system. These notebooks should be permanently bound record books or loose-leaf binders. If the latter is used, then all pages must be numbered sequentially so that removal of pages can be identified. The instrument should be identified with a unique name (with reference to the name used in the LIMS if not the same). The

logbook should provide a complete record of instrument usage, including changes to locations or power connections, maintenance performed, and any operational parameters that document conditions that may affect the quality of data obtained. Together with the Method document that identifies the instrument, the logbook should provide a complete record of instrument conditions, calibrations, and events that may aid in diagnosing problems that affect data quality.

Performance of calibrations or the analysis of check standards, especially after a change to instrument conditions or software, or after an instrument repair are especially important to document. All instrument malfunctions and repairs should be recorded. Analysts may record job numbers or sample names in logbooks, although this information should be adequately captured in instrument data files.

Quality Control

Quality Control (QC) samples shall be analyzed periodically to check method performance, instrument operation, and potential contamination. These samples may consist of standards, duplicates, or blanks and must be named according to documentation provided by the QC Lead. Results of the analysis of QC samples shall be submitted to the LIMS at the same time as corresponding analytical results on submitted samples. It is acceptable practice for the Analyst to determine that instrument operation or method performance is unacceptable based on analysis of QC samples prior to submittal of analytical results on submitted samples. In this case, the Analyst shall document results of QC samples in the instrument logbook and notify the appropriate section lead of the condition. Any suspect analytical results in the LIMS shall be left unvalidated and no analytical results shall be submitted until instrument operation and (or) method performance is judged acceptable. If suspect analytical results have been validated by the Analyst prior to identifying the condition, then the QC Lead shall be notified to not approve the suspect results. If any suspect results have been released, then the QC Lead shall notify the QA Lead of the condition so that any suspect data that may have been released can be tracked by initiating a concern.

Data Backup

Every instrument that stores acquired data shall have a backup scheme that is documented in either the instrument logbook or in method documents. The documentation shall include the logical location (i.e. data path) of the acquired data and acquisition parameters and the scheme by which these locations are backed up on a periodic basis. If a script or code is used to perform the backup, then this script or code should be listed in the documentation. Periodic backups may be performed either manually or automatically and the frequency of the backup listed in the documentation.

Data Traceability

Traceability of data requires that LIMS assigned sample names be maintained throughout the analytical process, although some methods may require additional suffixes to be added to assigned names. If instrument data acquisition software precludes the use of assigned names, then the instrument logbook shall show a listing of assigned names along with the names utilized by the analysis software. In all cases, it shall be possible to trace acquired data and processed data to results in the LIMS.

8. Samples

Proper collection, storage, and transport of samples are important to ensure the integrity and representativeness of sampled materials. Packaging must be sufficient to prevent leakage or inadvertent contamination. Because the sampling activities occur outside the scope of the EGL and involve other personnel, many of these sample issues are not covered by this QA Manual. The specifics of sample handling that occur after the samples arrive at the EGL facility are covered in EGL Method 25, Sample Acceptance and Registration [Log-In].

9. Issues, Concerns, and Corrective Action

Issues, concerns, and complaints from customers, Analysts, or audits shall be compiled, tracked, and responded to by the Lab Manager. An email address for issues or concerns has been established: EGL_qualityissues@usgs.gov. Emphasis of this system shall be on correcting the data (with appropriate audit trails in the LIMS) and on establishing or revising procedures to prevent recurrence of the same problem.

A database maintained by the Lab Manager shall track:

- Unique ID assigned to each issue or concern
- Assigned severity
- Job number(s) and sample(s) affected
- Title and text of concern
- Action taken to correct and to prevent recurrence
- Dates of record and status
- Personnel involved in reporting, recording, and responding

A form (Attachment 3) will be printed for each closed issue or concern; these forms will be kept by the Lab Manager and made available on request to staff or to auditors.

10. Records

Records generated by the EGL include:

- Instrument Logbooks
- Laboratory Notebooks
- Instrument data files and data formatted for input to the LIMS

- LIMS Database
- EGL Complaints Database and Closure Form (Attachment 3)
- Documentation of Training and Method Review forms (Attachments 1 and 2)
- Reports of audits and surveillances

The Laboratory Information Management System (LIMS) tracks who, what, when, and how data are obtained. All current data are irrevocably linked to an Analyst, instrument, and Method. Data are typically acquired from an instrument, processed by the Analyst using algorithms detailed in a Method, formatted into files suitable for LIMS input, and then uploaded to a specific network location. After the files are uploaded to the LIMS, the Analyst checks the data for correctness and validates the data. Upon validation, all subsequent changes to data shall generate an audit trail within the LIMS.

Prior to release of data, the QC Lead checks all relevant QC samples and then approves the data for release to the customer. Data entry, validation, and approval functions shall be tracked within the LIMS.

Protection of Data

The LIMS database shall be stored in a separate building and backed up onto separate media on a regular basis.

Instrument data shall be stored on local computers and shall be backed up to either local external media or to network locations.

Current laboratory notebooks and instrument logbooks shall be stored in the laboratory, but archived notebooks and logbooks shall either be stored locally in a fire-resistant cabinet or stored off-site.

11. Audits, Surveillances and Tests

Audits may be scheduled at regular intervals or requested on an irregular timeframe by the ERP Coordinator or designee. The scope of the audit shall be defined in advance to indicate which QA Manual policies or Methods are subject to the audit. Audits are generally performed by outside personnel. Written reports of audits shall contain the scope of the audit and any findings or recommendations.

Surveillances may occur at any time in order to verify compliance with QA Manual and established Methods, usually requested by QA Lead. A surveillance consists of an evaluation of the effectiveness of a QA policy or a Method to determine if the policy or Method is being implemented and to provide suggestions for improvements in a policy or Method that would enhance data quality and (or) traceability. Written reports of surveillances shall contain the scope of the surveillance and any findings or recommendations. Surveillances may be performed by EGL staff or outside personnel.

Findings from audits and surveillances shall be entered into the EGL Complaints database and tracked to closure.

Tests such as “blinds” shall be performed periodically and results reported to the QA Lead.

12. Approvals

The QA Manual shall be approved by the QA Lead and the Science Center Director.

Upon approval, EGL staff shall be issued a reading assignment to read the QA Manual and shall indicate completion by a signed training form (Attachment 1).

13. Revision History

Version 2.0

Major revision to the QA Manual, including the addition of sections on Requirements; Document Control; Methods; Issues, Concerns, and Corrective Action; and Audits, Surveillances and Testing. The Manual Overview section is expanded into a numbered section, section one in version 1.2 is revised to include an expanded section on Roles and Responsibilities, and portions of the QA/QC Overview subsection have been deleted, although portions are retained in the sections on Methods and Audits, Surveillances, and Testing. Records is now a stand-alone section, and section two in version 1.2 is handled under Methods; Audits and Corrective Action have been elevated to stand-alone sections. Portions of the manual dealing with safety and ethics have been removed from this version. With the exception of the logbook requirements, the instrumentation section is now part of Methods. The section on samples is handled by its own Method.

Record of Training Form

Name

Date of Completion

Course Duration

Type of Training

- Classroom
- Computer-based
- Reading

Course Title

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Course Description

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Course Organization

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Method Approval Form

Method Number **Revision Number**

Title
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.....

Previous Title
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.....

Primary Author _____

Secondary Author _____

Technical Reviewer _____

Add'l Technical Reviewer _____

QC Lead _____

QA Lead _____

Science Center Director _____

Approval Date