



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L802

Quality Control and Quality Assurance

Standard Operating Procedure

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1. Purpose

This SOP establishes the overarching quality assurance (QA) and quality control (QC) framework for all analytical work in the EA Program laboratory. It defines the minimum QC requirements that apply across methods, describes how to calculate and interpret QC metrics, specifies detection limit procedures, and establishes the data documentation and reproducibility practices required for every analysis. Individual analytical SOPs reference this document for general QC principles and add method-specific requirements (APHA et al., 2012; U.S. Environmental Protection Agency, 2007).

Quality assurance is the system of management actions that ensures the analytical process produces results of known and documented quality. Quality control is the set of technical activities (blanks, standards, duplicates, spikes) embedded in each analytical batch to measure and control that quality in real time (Taylor, 1987).

2. Scope

- **Applies to:** All quantitative chemical, physical, and biological analyses performed in the EA Program laboratory, including those described in SOPs L002 through L999 and analyses of field-collected samples (SOPs F001 through F999)
- **Does not apply to:** Qualitative screening tests (e.g., visual soil texture by feel, [SOP-F401](#)) unless those tests include a quantitative component
- **Location:** EA Program laboratory, SGM 133

3. Responsibilities

Role	Responsibility
Lab PI (QA Manager)	Approve this SOP; review QC data quarterly; authorize corrective actions
Lab Manager	Maintain instrument logs, reagent inventory, and QC records; train new analysts
Analyst	Implement all QC requirements for each batch; flag QC failures; document corrective actions
Data Reviewer	Verify QC compliance before data are reported or published

4. Definitions

- **Accuracy:** The closeness of a measured value to the true or accepted reference value; assessed using certified reference materials (CRMs) and laboratory-fortified blanks (LFBs) (Taylor, 1987).
- **Calibration blank:** A solution containing only the analytical solvent or reagent matrix (no analyte); used to establish the instrument baseline (zero) for each calibration sequence.

- **Certified reference material (CRM):** A material with certified analyte concentrations traceable to a national or international metrology institute (NIST, USGS, IRMM); used to verify analytical accuracy across instruments and over time (APHA et al., 2012).
- **Control chart (Shewhart chart):** A time-series plot of QC sample results with control limits (typically ± 2 warning limits and ± 3 action limits); used to distinguish random analytical variation from systematic drift or instrument malfunction.
- **Continuing calibration verification (CVS):** A mid-range calibration standard analyzed at regular intervals (typically every 10 samples) during an analytical run; must recover within $\pm 10\%$ of the stated value; failure requires re-calibration and re-analysis of affected samples (APHA et al., 2012).
- **Detection limit – Method Detection Limit (MDL):** The minimum concentration of an analyte that can be detected with 99% confidence under stated method conditions; determined from at least 7 replicate analyses of a low-level spike following 40 CFR Part 136 Appendix B (U.S. Environmental Protection Agency, 2016).
- **Detection limit – Limit of Blank (LOB):** The highest signal expected from a blank sample; $LOB = \text{mean}_{\text{blank}} + 1.645 \times SD_{\text{blank}}$ (Armbruster & Pry, 2008).
- **Detection limit – Limit of Detection (LOD):** The lowest analyte concentration that can be reliably distinguished from the LOB; $LOD = LOB + 1.645 \times SD_{\text{low-concentration}}$; represents 95% detection probability (Armbruster & Pry, 2008).
- **Detection limit – Limit of Quantitation (LOQ):** The lowest concentration at which the analyte can be measured with acceptable precision and accuracy, typically defined as $10 \times SD$ at the LOD spike concentration; also known as the practical quantitation limit (PQL) (Armbruster & Pry, 2008).
- **Field blank:** A sample of reagent-grade water processed through the entire field collection and preservation procedure; detects contamination introduced during sampling and transport.
- **Laboratory-fortified blank (LFB):** A reagent water blank spiked with a known concentration of target analytes and processed through the full analytical procedure; monitors ongoing accuracy and extraction efficiency.
- **Laboratory-fortified matrix (LFM):** A field sample spiked with a known concentration of target analytes; detects matrix-specific suppression or enhancement of the analytical signal.
- **Method blank:** A reagent water sample carried through the full analytical procedure alongside field samples; detects contamination introduced by reagents, equipment, or laboratory environment (APHA et al., 2012).
- **Precision:** The reproducibility of repeated measurements of the same sample; expressed as relative percent difference (RPD) for duplicates or relative standard deviation (RSD) for replicate analyses (Taylor, 1987).
- **Relative percent difference (RPD):** $|A - B| / ((A + B) / 2) \times 100$; the standard measure of duplicate precision; typical acceptance criteria are RPD 10–20% depending on method and matrix.
- **Spike recovery (%):** $(\text{Measured spiked concentration} - \text{Unspiked concentration}) / \text{Added concentration} \times 100$; typical acceptance range is 80–120% for most environmental matrices (APHA et al., 2012).

5. Safety and Hazards

QC samples contain the same analytes as field samples and are subject to the same hazard considerations as the parent analytical method. Consult the relevant analytical SOP for safety information.

6. General QC Requirements

6.1. Every Analytical Batch

Every analytical batch must include at minimum (APHA et al., 2012; U.S. Environmental Protection Agency, 2007):

QC Element	Minimum Frequency	Purpose
Method blank	1 per batch	Detect laboratory contamination
Calibration blank	Start of each calibration	Instrument baseline
CVS (check standard)	Every 10 samples	Detect calibration drift
Duplicate	10% of samples	Assess precision
LFB	1 per batch	Assess accuracy

6.2. Additional Elements (as applicable)

QC Element	When Required
Field blank	Each sampling event
Matrix spike (LFM)	5% of samples or 1 per batch
CRM	1 per batch (where certified material is available)
Equipment blank	When non-dedicated sampling equipment is used

7. Acceptance Criteria

QC Element	Typical Acceptance Criteria	Action if Failed
Method blank	< MDL (analyte absent)	Investigate contamination; do not report affected samples
CVS	$\pm 10\%$ of stated value	Re-calibrate; re-analyze affected samples
Duplicate RPD	10–20% (method-specific)	Flag results; investigate and re-analyze if warranted
LFB recovery	80–120%	Flag results; check reagents and instrument

QC Element	Typical Acceptance Criteria	Action if Failed
LFM recovery	70–130%	Suspect matrix interference; consult PI
CRM recovery	Within certified uncertainty interval	Recalibrate; re-analyze

Acceptance criteria in individual analytical SOPs take precedence over these general values when they are stricter or when regulatory requirements apply.

8. Detection Limit Determination

8.1. Method Detection Limit (MDL)

The MDL is determined following 40 CFR Part 136 Appendix B (U.S. Environmental Protection Agency, 2016):

1. Select a spike concentration at $1\text{--}5\times$ the estimated MDL.
2. Prepare and analyze at least 7 replicates of the spiked solution through the full analytical method.
3. Calculate the standard deviation (s) of the replicates.
4. Multiply by the one-tailed t -value at 99% confidence ($t_{n-1,0.99}$; for $n = 7$: $t = 3.143$):

$$\text{MDL} = t_{(n-1, 0.99)} \times s$$

5. If the spike concentration is outside the range of $0.5\text{--}20\times$ the computed MDL, repeat with an adjusted spike concentration (U.S. Environmental Protection Agency, 2016).

8.2. Limit of Detection and Limit of Quantitation

The LOB, LOD, and LOQ hierarchy follows Armbruster & Pry (2008):

$$\text{LOB} = \mu_{\text{blank}} + 1.645 \sigma_{\text{blank}}$$

$$\text{LOD} = \text{LOB} + 1.645 \sigma_{\text{low}}$$

$$\text{LOQ} = 10 \sigma_{\text{low}}$$

where σ_{low} is the standard deviation of replicate analyses at the low-level spike concentration. For right-censored environmental data, apply the non-parametric methods described in Helsel (2012).

9. Control Charts

Maintain control charts for each analyte/method combination to track long-term analytical performance:

1. Plot CRM, LFB, or CVS recovery for each batch on a time-series chart.
2. Calculate and draw lines for:
 - **Center line:** Mean recovery over the last 20+ batches
 - **Warning limits:** ± 2 standard deviations from the mean
 - **Action limits:** ± 3 standard deviations from the mean
3. A result between warning and action limits is a warning; investigate but do not necessarily re-analyze.
4. A result beyond the action limits requires corrective action and re-analysis.
5. Systematic trends (7+ consecutive results on one side of the center line, or a steady drift) require investigation even if individual points are within limits (Taylor, 1987).

10. Corrective Actions

When a QC failure occurs:

1. Stop the analytical run; do not report affected samples.
2. Identify the probable cause (contaminated blank, degraded standard, instrument malfunction, matrix interference).
3. Correct the problem (replace reagents, recalibrate, perform maintenance).
4. Re-run the affected samples with fresh QC elements.
5. Verify that the repeated run passes all QC criteria.
6. Document the failure, root cause, corrective action, and re-analysis results in the QC log and lab notebook.

11. Metadata Documentation and Reproducibility

11.1. Laboratory Notebook Requirements

Every analytical session must be documented with at minimum:

- Date, analyst name, SOP number and revision
- Instrument ID and calibration status
- Reagent lot numbers and preparation dates
- Sample IDs, matrix type, and collection dates
- Calibration data (standards, equation, r^2)
- QC results (blanks, duplicates, LFBs, spikes, CRMs) with pass/fail
- Raw data file names and storage location
- Any deviations from the SOP, with justification
- Corrective actions taken for QC failures

11.2. Electronic Data Management

1. Store all raw instrument data files on the lab shared drive in a folder structure organized by project, year, and instrument.
2. Never modify raw data files. All processing must be done programmatically in R with version-controlled scripts.
3. Commit all analysis scripts to the lab GitHub repository per [SOP-L801](#) (Gandrud, 2015; Xie, 2015).

11.3. R-Based QC Tracking System

The following R framework is the EA Program standard for QC documentation and reproducibility (Gandrud, 2015; Xie, 2015):

```
library(tidyverse)
library(lubridate)
library(knitr)

session_meta <- list(
  analyst   = "Your Name",
  date      = Sys.Date(),
  sop       = "SOP-L802 v1.1",
  method_sop = "SOP-LXX vX.X",
  instrument = "Instrument ID",
  project   = "Project Name"
)

cat(sprintf("QC session: %s | Analyst: %s | Instrument: %s\n",
            session_meta$date, session_meta$analyst,
            session_meta$instrument))
```

```
calc_mdl <- function(reps, alpha = 0.01) {
  n <- length(reps); s <- sd(reps)
  t_val <- qt(1 - alpha, df = n - 1)
  list(n = n, sd = s, t_value = round(t_val, 3),
       MDL = round(t_val * s, 4), LOQ = round(10 * s, 4))
}

calc_rpd <- function(a, b) abs(a - b) / ((a + b) / 2) * 100

calc_recovery <- function(measured, expected) (measured / expected) * 100
```

Each analytical R script should:

1. Read the raw data file
2. Run all QC checks programmatically

3. Produce a formatted QC summary table
4. Generate calibration plots and control charts
5. Export a clean, QC-verified dataset as CSV
6. Render to HTML or PDF for archiving

Store templates in the project GitHub repository under `analysis/templates/` (Gandrud, 2015; Xie, 2015).

12. QC Requirements by Method Category

QC Element	Water Chemistry	Soil Extraction	Digestion (Metals)	Biological (Colilert)	Instrumental (TOC, ICP)
Calibration blank	Each cal	Each cal	Each cal	N/A	Each cal
Method blank	1/batch	1/batch	1/batch	1/batch	1/batch
Field blank	1/event	N/A	N/A	N/A	N/A
Duplicate	10%	10%	10%	10%	10%
LFB	1/batch	1/batch	1/batch	N/A	1/batch
Matrix spike	5% (or 1/batch)	5%	5%	N/A	5%
CRM	1/batch (if avail.)	1/batch	1/batch	Positive control	1/batch
CVS	Every 10 samples	Every 10	Every 10	N/A	Every 10

13. Waste Disposal

QC samples generate the same waste streams as routine samples. Dispose of QC waste (blanks, standards, spikes) according to the parent method's waste disposal procedure.

14. References

- APHA et al. (2012)
- Taylor (1987)
- U.S. Environmental Protection Agency (2007)
- U.S. Environmental Protection Agency (2016)
- Armbruster & Pry (2008)
- Helsel (2012)
- Gandrud (2015)
- Xie (2015)

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- Armbruster, D. A., & Pry, T. (2008). Limit of blank, limit of detection and limit of quantitation. *Clinical Biochemistry Reviews*, 29(Suppl 1), S49–S52.
- Gandrud, C. (2015). *Reproducible research with R and RStudio* (2nd ed.). CRC Press.
- Helsel, D. R. (2012). *Statistics for censored environmental data using Minitab and R* (2nd ed.). John Wiley & Sons. <https://doi.org/10.1002/9781118162729>
- Taylor, J. K. (1987). *Quality assurance of chemical measurements*. Lewis Publishers.
- U.S. Environmental Protection Agency. (2007). *Guidance for quality assurance project plans* (EPA/240/R-02/009). Office of Environmental Information.
- U.S. Environmental Protection Agency. (2016). *Definition and procedure for the determination of the method detection limit, revision 2* (40 CFR Part 136, Appendix B). U.S. Environmental Protection Agency. <https://www.epa.gov/cwa-methods/mdl-determination-procedure>
- Xie, Y. (2015). *Dynamic documents with R and knitr* (2nd ed.). CRC Press.

15. Related SOPs

SOP	Title	Relationship
L801	RStudio and GitHub	Version control for analysis scripts
L803	Data Entry and Database	Data recording standards
F300	Surface Water Sampling	Field blank requirements
F008	Sample Labeling and COC	Chain of custody

16. Training Record

Before applying QC frameworks independently, personnel must:

1. Read this SOP in full
2. Demonstrate correct MDL calculation using the `calc_mdsl()` function on a test dataset
3. Prepare a complete QC summary table for one analytical batch under supervision
4. Set up and interpret a control chart for one analyte/method combination
5. Be signed off by the PI or lab manager