ALTA

AGRICULTURAL LABORATORY TESTING ASSOCIATION

Overview:

- **Illinois Soil Testing Association (ISTA) was founded in 1981 address Illinois growers' needs for quality soil test information. ISTA rebranded as the Agriculture Laboratory Testing Association (ALTA).**
- **ALTA's mission is to promote the interests of the Ag testing industry and advance high-quality soil & plant-tissue analysis data for farm profitability, and sustainability in the US.**
- **ALTA is committed to ensuring the quality of data to agricultural communities by encouraging the development, use, and acceptance of proven agricultural testing methods.**

Laboratory Quality Management - Basics of QC

Robert O. Miller, PhD
ALP Technical Director
Fort Collins, CO

Rmiller@soiltesting.us

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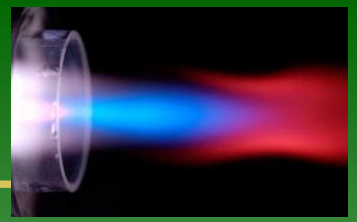
LQM Overview



Lab quality management (LQM) is the aggregation of all lab activities including management associated with the generation of reliable analytical test measurement data.

A LQM system is comprised of two primary components Quality Assurance (QA) and Quality Control (QC).

Overview

Quality Assurance (QA), as defined by ISO is *"the assembly of all planned and systematic actions necessary to provide adequate confidence that product, process or service will satisfy given quality requirements".*

Quality Control (QC), is defined as *"the operational techniques and activities that are used to satisfy quality requirements" within a method SOP (Standard Operating Procedure).*

Quality Control

QC is a monitoring program undertaken specifically to achieve accurate and reliable analytical results and the prevention of errors. It is a means to document the uncertainty and the ongoing performance of an analytical method and assure reliable performance.

Within the SOP, QC constitutes those operations that monitor and verify method performance, to assure reliable results.

Quality Control

Method SOP

QC batch element(s)

Calibration
Method blank
Duplicate
Matrix spike
Reference material

QC Process monitoring

MDL, LOQ
Analyte carry-over
Control charts
Instrument log
Proficiency testing

Method SOP QC batch

The method SOP QC identifies the quality batch element, frequency and acceptance criteria:

- Instrument Calibration (IC)
- Calibration Blank (CB)
- Method Blank (MB)
- Continuing Calibration Verification (CCV)
- Independent Calibration Verification (ICV)
- Interference Check Solution (ICS) *optional*
- Duplicate (DUP)
- Matrix Spike Recovery (MSR)
- Certified Reference Material (CRM)

[1] *This QC list is not inclusive all components, maybe method specific*

SOP QC batch elements

Instrument Calibration (IC). Initial calibration defines the number of calibration standards, minimum and maximum concentration, regression type and minimum coefficient of determination.

Calibration Blank (CB): Sample/solution matrix matched with calibration standards and is used for initial and continuing instrument calibration.

Method Blank (MB). The method blank is used to test for contamination of sample preparation process, carried through the entire analysis procedure including extraction / digestion.

SOP QC batch elements (cont)

Continuing Calibration Verification (CCV). CCV solution(s) used for initial and continuing verification of instrument calibration prepared from stock standard solutions and used assess error between known and measured concentrations.

Independent Calibration Verification (ICV). One ICV per batch, prepared from a source other than that which the calibration stock standards are prepared.

Duplicate (DUP). A duplicate unknown sample should be processed with each batch and separated in the analytical batch from the original sample. Measured as Relative Percent Difference (RPD).

SOP QC batch elements (cont)

Matrix Spike Recovery (MSR). A known quantity of analyte is added to an unknown sample, and percent recovery is calculated. Spike concentration approximately 100 x MDL.

Certified Reference Material (CRM). A matrix similar to that of materials tested should be obtained to assess the accuracy and overall reliability of the analytical process and analyzed with each batch. Alternate non-certified Standard Reference Material (SRM).

SOP QC Process monitoring

Method Detection Limit (MDL). Low level method blank spike evaluated for determining detection limit and Limit of Quantification (LOQ), bi-annually.

Analyte Carry-Over (ACO). Potential error associated with automated analyzers when residual analyte carries over into the next sample. Evaluated based on schedule (e.g. bi-annually, annually).

Control Charts (CC). Record batch results of MB, MSR, and CRM, plot over a timeline. Range charts (RC) are timeline plots of duplicates.

Instrument Log (IL). Document log of instrument performance, maintenance service and repairs.

Proficiency Requirement (PR). Laboratory shall participate in a proficiency testing program compliant with method SOP. Evaluated biannually or tri-annually.

SOP QC: purpose

The method SOP QC defines the batch element purpose, function and brief discussion of implementation.

Method Blank (MB) [1]. The method blank is used to test for contamination during the sample preparation process. The MB should be carried through the entire sample analysis procedure including extraction / digestion / analysis. The MB must contain all of the reagents in the same volumes used in the processing of the samples.

[1] Example from, Hicks, K. 2022. Inductively coupled plasma spectroscopy analysis. Recommended Methods of Manure Analysis, 2nd ed. (in press).

SOP QC: frequency

The method SOP defines the frequency of QC monitoring and is typically based on the analytical “batch” size of unknown samples.

Batch size can be a single unknown sample, or an array of 10, 20 or 50 unknowns samples, dependent on:

Specific analyte
Method stability
Required measurement uncertainty

QC process monitoring, such as MDL, ACO, ACO, IL, CC and PR, may be based on a systematic schedule (e.g. hourly, daily, annually).

SOP QC: acceptance criteria

The method SOP QC defines acceptance criteria and specify steps to resolve a failure.

Method Blank (MB) [1]. The method blank is used to test for contamination during the sample preparation process. The MB should be carried through the entire sample analysis procedure including extraction / digestion / analysis. The MB must contain all of the reagents in the same volumes used in the processing of the samples.

Acceptance criteria: Concentrations of the measured analyte must be below the MDL or within documented historical acceptance limits. If criteria is exceeded, re-analyze MB. If 2nd MB passes, calibration is verified. If 2nd MB fails, halt analysis until source of contamination eliminated.

[1] Example from, Hicks, K. 2022. Inductively coupled plasma spectroscopy analysis. Recommended Methods of Manure Analysis, 2nd ed. (in press).

SOP QC: acceptance criteria (cont)

Independent Calibration Verification (ICV)[1]. One ICV shall be analyzed with each analytical batch. The ICV should be prepared from a source other than the one from which the calibration stock standards are prepared. Calculate the percent error (PE) between the ICV known concentration and as-measured concentration.

$$PE = \frac{known\ value - measured\ value}{known\ value} \times 100$$

Acceptance criteria: Ensure that results are within ± 10 PE. If criteria is exceeded, re-analyze ICV. If 2nd ICV passes, calibration is verified. Otherwise recalibrate with new calibration standards and re-analyze all samples since last valid ICV.

[1] Example from, Hicks, K. 2022. Inductively coupled plasma spectroscopy analysis. Recommended Methods of Manure Analysis, 2nd ed. (in press).

SOP QC example: manure NH_4-N

QC batch element [1]	Frequency	Acceptance criteria
Calibration	Every batch	*$r^2 \geq 0.99$ for linear and $r^2 \geq 0.999$ for polynomial*
Calibration Blank (CB)	After calibration, every 10 samples	NH_4 -N < MDL
Method Blank (MB)	Every batch	NH_4-N < LOQ
Continuing Calibration Verification (CCV)	After calibration, every 10 samples	Within ± 10 PE
Independent Calibration Verification (ICV)	After calibration	Within ± 10 PE
Duplicate (Dup)	Every batch	RPD ≤ 10% within the documented acceptance limits
Matrix Spike Recovery (MSR)	Every batch	Recovery, 100 ± 15%
Standard Reference Material (SRM)	Every batch	Within uncertainty limits

[1] Example from: Miller, R.O., K. Hicks, J. Lessl, J. Spargo, and J. Mowrer. 2022. Nitrogen, ammonium (NH_4-N) by distillation, spectrophotometry, and diffusion-conductivity. Recommended Methods of Manure Analysis, 2nd ed. (in press).

SOP QC –example

Plant total phosphorus (P) analysis

Sample dried / pulverized, 0.50 g microwave digestion with HNO_3, 25 mL final dilution volume (DF = 50), 24 digestion vessels.

Analysis by Thermo Fisher 6500 ICP-OES crossflow nebulizer, radial view, wavelength 177.4 nm, internal yttrium standard. Analytical batch size n = 20 unknown samples.

Method SOP QC batch requirement: calibration, CB, MB; CCV; ICV; Dup; MSR and CRM.

Method QC process monitoring: MDL, LOQ, AOC, CC, IL and PR.

SOP QC batch – total P

QC batch element	QC Application	Acceptance criteria
Calibration	Six P standards 1.0, 2.0, 5.0, 10, 20, 50 and 100 mg/L, matrix matched, 5% HNO_3	*$r^2 \geq 0.999$ for polynomial*
Calibration Blank (CB)	RGW, matrix matched 5% HNO_3	P < MDL
Method Blank (MB)	Microwave digest blank, one per batch	P < LOQ
Continuing Calibration Verification (CCV)	CCV repeated every ten samples, concentration 20 x LOQ, matrix matched	Within ± 10 PE[2]
Independent Calibration Verification (ICV)	External sourced ICV sample 50 mg/L, one per batch	Within ± 10 PE
Duplicate (Dup)	Random selected duplicate of unknown sample, calculate Dup RPD	RPD ≤ 10% within the documented acceptance limits
Matrix Spike Recovery (MSR)	Spike of unknown sample with P concentration 20-40% above estimated P content	Recovery, 100 ± 15%
Certified Reference Material (CRM)	NIST CRM, P content 2040 ± 120 mg/kg	Within uncertainty limits

[1] *Method MDL 0.21 mg/kg, LOQ 0.65 mg/kg.*
[2] *Percent error, (known value – measured) / (known value).*

SOP QC method monitoring

QC monitoring	Application	Schedule
Method detection limit	MDL determined in accordance of US-EPA 821-R-16-006	Biannually, or after instrument repair
Assessment of Analyte-Carry Over	Three high standards followed by three blanks, Method of Dixon 1990	Annually, or after instrument repair
Control charts	Control chart graphs of CRM mean charts and dup range charts	Every batch
Instrument log	Record of instrument performance, maintenance and repairs	Daily/Weekly
Proficiency Requirement	Analysis and method SOP matched	Tri-annually

Determining QC batch size

Analytical batch size is typically configured based on lab workload, labware configurations (i.e. digestion block, extraction racks, autoloaders), manageable unit, process / instrument stability and/or cost.

Batch size maybe limited due to:

SOPs with time sensitive procedures or steps

Analysis / instrumentation susceptible to drift

Instrumentation with long analysis times, minutes

Recommendation. Evaluate method SOP sample preparation, procedural steps and instrument stability prior to establishing analytical batch size.

Summary

The method SOP QC is essential for monitoring and the documentation of analytical results.

The SOP QC is divided into two units, those associated with the analytical batch and those with method monitoring.

The primary QC batch elements are: calibration, calibration blank, method blank, continuing calibration verification, independent calibration verification, duplicate, matrix spike recovery and certified reference material.

Special thanks

John Spargo, Associate Research Professor &; Director, Agricultural Analytical Services Lab. Penn State University, jts29@*psu*.edu

Kristin A. Hicks, Section Chief, Plant/Waste/Solution, NCDA&CS Agronomic Division, kristin.hicks@ncagr.gov

Jason Lessl, Assistant Professor University of Georgia Soil, Plant, & Water Lab, jlessl@uga.edu

Patrick Visser**,** Vice President, Farmers Edge Laboratory, Manitoba, Canada, Patrick.Visser@FarmersEdge.ca

References

EPA, 1986. Quality Control. In: Test Methods for Evaluating Solid Waste, Vol. 1A: 3rd ed. EPA/SW-846,. National Technical Information Service. Springfield, Va.

Garfield, F. M. 1991. Quality Assurance Principles for Analytical Laboratories. Association of Official Analytical Chemists. Arlington, VA.

Ogden, L., P. Kane, P. Knapp, N. Thiex, L. Torma, 1998. Association of American Feed Control Officials Quality Assurance/Quality Control Guidelines for State Feed Laboratories. Association of American Feed Control Officials.

Thiex, N., L. Torma, M. Wolf, and M. Collins, 1999. Quality Assurance Quality Control Guidelines for Forage Laboratories. National Forage Testing Association.

van Reeuwijk, L.P. 1998. Guidelines for Quality Management in Soil and Plant Laboratories. FAO Soils Bulletin 74. ISRIC, Rome, Italy.

Laboratory Quality

Quality is …….

- **invisible when its GOOD**
- **impossible to ignore when its BAD**

ALTA-SPAC Webinars

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Date	Webinar Title
January 26, 2021	The Laboratory Quality Management- Preparing for 2021
March 25, 2021	The Laboratory Standard Operating Procedure (SOP)
July 27, 2021	Determining the Method Detection Limit (MDL)
August 24, 2021	Soil Scooping: Assumptions & Issues I
January 18, 2022	Soil Scooping: Assumptions & Issues II
March 29, 2022	Laboratory Quality Management - Basics of QC

Visit: alta.ag/presentations

ALTA-SPAC Webinar - June 2022

Topic:

"Overview of *Recommended Methods of Manure Analysis*, 2nd Edition"

Date: TBA

Thank you for your time and attention

E-mail:
RMiller@soiltesting.us
Txt: 970-217-2572

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