

Overview:

- Illinois Soil Testing Association (ISTA) was founded in 1981 to help address Illinois growers' needs for quality soil test information.
- In Dec. 2020, ISTA rebranded as the Agriculture Laboratory Testing Association (ALTA).

ALTA's Purpose:

 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.
- Our goal is to be the industry leader in ensuring consistency, precision, and accuracy across Ag laboratories nationally, through outreach, education, and certification programs.

2021 Membership:

- 21 current member laboratories located primarily in the Midwest.
- New for 2021 is the ALTA–PAC for certification of plant analysis.

The Laboratory SOP

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

Rmiller@soiltesting.us

ALTA Webinar March 25, 2021





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

Fundamental to the LQM is the laboratory Standard Operating Procedure (SOP).





The SOP document defines a compulsory set of instructions for regular recurring operations and processes of the laboratory, to ensure they are carried out in consistent manner.

A detailed SOP defines the scope of the method or process, that ensures method execution, minimizes variability in results, and helps in resolving deviations in analytical results.





Most are familiar with the analytical method SOP, but SOPs may span all lab aspects of lab processes and operations.

Sample receipt/login Cleaning of labware Reagent storage Hazardous waste disposal Standard preparation Staff training Lab administration LIMS operation Report generation Data archival Staff training Client complaints and instructions on the preparation of an SOP.



The SOP method outline

Scope and application method / process Principle of the method / process Terminology / Definitions Personnel - Training Sample and Prep Instruments / equipment Reagents Procedure Reporting **Quality Control** Health and Safety Reference documents







Components of a method SOP



Title	Scope	Samples	Roles Training	Equipment Reagents	Procedure	Report	QC	Safety	Ref Docs
-------	-------	---------	-------------------	-----------------------	-----------	--------	----	--------	----------

Title: provides reader with subject, method or process

Description: method or process; describes performance, interferences, stability

Sample Prep: covers sample processing, handling stability method or process

Personnel: staff qualifications and training

Materials: Instruments, labware and chemicals

Process: step by step description

Report: reporting basis / units

QC: process quality control

Health and Safety

Ref Docs

Miller, 2021



ALTA Webinar The Laboratory SOP March 25, 2021

https://drug-dev.com/wp-content/uploads/2020/03/F1-6.jpg



Manure ammonium (NH₄-N): Spectrophotometric

1.0 Scope and Application

The method quantitatively determines the concentration of ammonium nitrogen (NH_4 -N) animal manures (liquid, semi-solid or solid) by extraction with potassium chloride (KCI) and subsequent analysis by spectrophotometry analysis. NH_4 -N concentrations are frequently requested by waste operators for regulatory compliance, nutrient management plans, permitting or when accidental discharges occur.

¹ Example list , additional issues may be laboratory specific.



SOP method example



Manure ammonium (NH₄-N): Spectrophotometric

2.0 Method Principle

2.1 <u>Method Description</u>. The ammonium nitrogen (NH₄-N) content of manure is determined by quantitative extraction using 1.0 <u>N</u> KCl and subsequent analysis by spectrophotometry. The method has been adapted from US-EPA 1690.

2.2 <u>Method Performance</u>. The user should be able to attain a Limit of Quantification (LOQ) of 0.0005 % NH_4 -N for an applicable calibration range of 0.005 – 0.500 %. Method uncertainty, concentration 0.20 ± 0.03 % NH_4 -N, on dry basis.

2.3 <u>Method Interferences</u>. For spectrophotometry samples containing materials that absorb light can interfere with method accuracy. Samples can be 'cleaned' by a dialyzer membrane.

2.4 <u>Sample Stability</u>. Samples shall be refrigerated at \leq 4 °C for 7 days prior to processing.

2.5 <u>Method Reporting</u>. For solid samples, NH_4 -N content is reported as % concentration, lbs ton⁻¹, or mg/kg reported on as-received basis .



SOP method example

3.0 Personnel and training

3.1 Staff training. Minimum knowledge, skill and abilities to conduct the method.

4.0 Sample Processing, Preservation and Storage

4.1 **Sample Processing**. Manure (slurry, semi-solid, or solid) samples containing less than an estimated 14% total solids shall be homogenized

4.2 <u>Sample Solid Content</u>. Determine as received Total Solid content according to Method M-X.10 and record.

4.3 Sample Requirements. Minimum sample is 1.0 g (s) and 10.0 ml (l).

4.4 Sample Stabilization.

4.5 <u>Sample Defrost</u>. Place frozen sample in refrigerator at \leq 6 °C for 48 hours.





Autorstock.com v 51452306

5.0 Equipment

- 5.1 Rotor-Stator Homogenizer. Max-Homogenizing unit minimum of 250 mL material.
- 5.2 Analytical Balance: minimum resolution required ± 0.01g
- 5.3 Storage extraction container
- 5.4 Reciprocating horizontal mechanical shaker
- 5.5 Büchner funnel filtration equipment

5.5.1 Glass fiber filter

5.6 <u>Technicon AutoAnalyzer II</u>, NH_4 -N analysis by alkaline phenol / sodium hypochlorite reaction with ammonia chemistry. Instrument detection limit 0.0001 % NH_4 -N.



6.0 Reagents



6.1 Deionized (DI) water. ASTM Type I grade, < 0.056 Conductivity at 25 $^{\circ}C/\mu S \cdot cm^{-1}$.

6.2 Extraction reagent. Potassium chloride (KCI) extracting solution, 1.0 N KCI: Dissolve 75 g of reagent grade KCI in 500 mL DI water and dilute to 1.0 L final volume with DI water .

6.3 Spectrophotometric Reagents

6.3.1 <u>Alkaline Phenol</u>. Dissolve 83 g phenol in a 1 L Erlenmeyer flask containing 500 mL deionized. While stirring, slowly add 32 gm NaOH. Cool, dilute to 1 L with DI water, and filter through a glass fiber filter if necessary. Store in a dark glass bottle.

6.3.2 Sodium Hypochlorite Solution: Dilute 500 mL of commercial bleach containing 5.25% available chlorine to 1 L with DI water, and filter if necessary. Store at \leq 6 °C.

6.3.3 <u>Buffer</u>. Dissolve 50.0 g disodium ethylenediamine-tetraacetate (Na₂ EDTA) and 12.5 g NaOH in 900 mL of DI water. Dilute to 1 L with DI water.

6.3.3 <u>Sodium nitroprusside</u>. Dissolve 7.0 g of $Na_2Fe(CN)_5NO \cdot 2H_2O$ in 900 mL of DI water and dilute to 1 L. Reagent is light sensitive, store in light proof container.



SOP method example

7.0 Procedure



7.1 Extract NH_4 -N from sample matrix.

7.1.1 <u>Slurry and Liquid waste</u>. Dilute 5:1 with 1 N KCl. 10-50 ml of liquid waste material. Cap and place on reciprocating shaker for 30 minutes. Filtered to remove particulate matter.

7.2 Spectrophotometric analysis.

7.2.1 Initialize instrument according to manufacturer instructions.

7.2.2 Monitor instrument baseline drift, run a Drift Correction Standard (DCS) using a midrange calibration standard followed by 1-2 rinse samples with DI water.

7.2.3 Perform a six-point calibration. The calibration range range of 0.5 - 200.0 mg NH_4 -N L⁻¹, evaluate slope and calibration.

7.2.4 Run one Method Blank 1 N KCL.

7.2.5 Run Independent Calibration Verification (ICV). Evaluate standard reference accuracy.

7.2.6 Analyze Matrix Spike Duplicate (MSD) once per analytical batch of 20 or fewer samples.

7.2.7. Analyze unknown sample(s), batch size maximum 20 samples.



SOP method example



8.0 Calculations and reporting

8.1 Unknown sample calculation

% NH_4 -N = [X_n × (V / m)] × D_f / 10000

where:

 X_n is the extract analyte primary result from instrument analysis in % V, total volume of extraction which includes unknown sample and KCI (ml) m, unknown sample mass (g or mL) D_f , additional dilution factors

Round NH₄-N results to three significant digits.

8.2 **Results less than LOQ**, report as not detectable (ND).

8.3 <u>Client Reports</u>. Shall report method reference, LOQ, RPD, reference standard result, spike recovery and method anomaly observation(s).



9.0 Quality Control



9.2 <u>Analysis batch requirements</u>: Calibration Blank (CB), Method Blank (MB), Continuous Calibration Verification (CCV) Independent Calibration Verification (ICV), Duplicate (D), Matrix Spike Recovery (MSR), and Reference Material (RM). Verify QC results within acceptance criteria.

9.3 **Proficiency Requirements**. Laboratory shall participate in a proficiency testing program for NH₄-N in manure materials that is compliant with International Laboratory Accreditation Cooperation (ILAC) G13 guidelines, minimum two rounds per year. Example, Minnesota Department of Agriculture, Manure Analysis Proficiency (MAP) Program





9.0 Limit of Quantification, Accuracy and Precision

10.1 Limit of Quantification (LOQ). Method Detection Limit (MDL) shall be determined based on based on the method described by the US-EPA, US-EPA 821-R-16-006. The LOQ shall be calculated by the following equation.

 $LOQ = MDL \times 3.0$

A LOQ less than or equal to 0.0005 % NH_4 -N must be achieved prior to the practice of this method. Determine MDL and LOQ when instrument is repaired or replaced.

10.2 <u>Method Performance</u>. NH_4 -N repeatability and reproducibility evaluated in accordance with ISO 5725-2 from results of the Manure Analysis Proficiency



11.0 Health and safety



11.1 <u>Health and Safety</u>. Manure can contain pathogenic organisms and compounds which cause eye or skin irritation. Always ware appropriate lab clothing, gloves, and eye protection.

11.2 <u>Laboratory Safety</u>. The laboratory is responsible for maintaining a current awareness file of state / federal regulations regarding the safe handling of the chemicals specified in this method. A Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis.

11.3 **<u>Reagents Disposal</u>**. Dispose of reagents in accordance with and local/state/federal regulations.

11.4 **Samples Disposal**. Dispose of manure in accordance with local/state/ federal regulations.

12.0 Reference documents

AOAC, Official Methods of Analysis of Association of Official Analytical Chemists, 18th edn., Arlington, VA, 1995, Protein (Crude) in Animal Feed: Combustion Method (990.3).

US-Environmental Protection Agency. 2001. Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses. Chapter 2. Document # EPA-823-B-01-002.



SOP process example

Soil grinding

- Scope and application of the process
- Principle of the process
- Equipment / manufacturer
- Process
- Training
- Maintenance schedule
- Documentation







The SOP document

Compar	ny Name, Inc.		Standard Operating Procedure			
Title:	aboratory Notebook Is	ssuance	Procedure Number: QASOP-xxx	Page Number: Page 1 of 3		
Function: Quality Assurance			Implementation Date:	Revision Number: 01		
Author: Signature	Name, Function	Date	Approved By: Name, Signature	Function Date		
Approved By: Signature	Name, Function	Date	Approved By: Name, Signature	Function Date		

1.0 PURPOSE

This procedure describes the issuance and retention requirements as well as the identification numbering system for laboratory notebooks at Company Name.

2.0 SCOPE

This procedure applies to all laboratory notebooks at Company Name.

3.0 DEFINITIONS

Documentation Administration – QA system designed to process, track, distribute, and file copies of controlled documents, including laboratory notebooks.

Laboratory Notebook (Lab-book) – Bound, legal documents in which are recorded scientific data, protocols, observations, schematics, ideas, and other information that is considered part of the company's research and development operations and intellectual property.

4.0 ASSOCIATED DOCUMENTS/REFERENCES

QAPOL-xxx – Documentation Policy

5.0 MATERIALS/EQUIPMENT

Laboratory notebooks (various models as appropriate)

6.0 SAFETY

There are no safety issues relevant to this document.

7.0 RESPONSIBILITIES

7.1 Quality Assurance is responsible for maintaining a record of all laboratory notebooks.

- 7.1.1 The records will track a unique identifying number for each lab-book.
- 7.1.2 The records will track who lab-books are issued to.
- 7.1.3 The records will track the subject/title of each notebook



The SOP document should identify:

The company The method Date generated Author(s) Approval signatures Length of operation Revision history



ALTA Webinar The Laboratory SOP March 25, 2021



Specific SOPs which require explicit method techniques or processes may require an instructional video. Examples:

- Soil scooping / dipping
- Preparation of a soil saturated paste
- Manure sample preparation
- Quantitative analytical transfers

A SOP video procedure can be an effective training tool.







The purpose of SOP is to document a set of instructions of a method or process.

It's purpose is to minimize ambiguity and ensure consistent execution of a method or process.

It defines the method or process and provides instruction for training.



SOP Resources



Guidelines for Quality Management in Soil and Plant Laboratories. (FAO Soils Bulletin - 74)

Authored by L.P. Reeuwijk Released at 2001

https://olinguru-ir.netlify.app/00-patience-dubuque-9/9788170352518guidelines-for-quality-management-in-soils-and-p-ebook.pdf



Guidelines for Quality Management in Soils and Plant Laboratories

Authored by L.P. Reeuwijk and Victor Houba Released at 2016



ALTA Webinar The Laboratory SOP March 25, 2021





Through this presentation there have been references made to the Method Detection Limit (MDL).

The MDL defines the minimum analyte concentration that can be determined. It is not the instrument detection limit (IDL).

A MDL webinar will be scheduled July 2021.



Thank you for your time and attention

E-mail: Robert.Miller@cts-interlab.com Txt: 970-217-2572

ΔΙ

AGRICULTURAL LABORATORY







The Plant Analysis Certification (PAC) program has launched sponsored by SPAC and ALTA. The program provides a standard of performance for plant mineral analyses. More information can be found at <u>www.ALTA.Ag</u>

