



Procedures Used by OSU Soil, Water and Forage Analytical Laboratory

Hailin Zhang
Laboratory Director

Kendal Henderson
Laboratory Coordinator

Oklahoma Cooperative Extension Fact Sheets
are also available on our website at:
facts.okstate.edu

The Soil, Water and Forage Analytical Laboratory (SWFAL), located in the basement of Agriculture Hall at OSU, was established to meet the soil, plant and water quality testing needs of farmers, ranchers, homeowners and consultants throughout the state. The laboratory provides fast and accurate analyses, and reliable interpretations, so the data obtained from testing can be used as a guide to maintain soil health and optimum crop production. The tests have been developed to assist producers in areas such as fertilizer requirements, forage quality, water quality, soil salinity assessment and animal manure nutrient contents.

Most tests offered have a firm research basis. This research involves many disciplines within OSU, and it is conducted at OSU Agronomy Research Stations and private cooperators' fields throughout the state. These ongoing studies, under diverse management and cropping systems, enable accurate, long-term calibration of each test.

Accurate laboratory results are maintained through the use of laboratory standards, blank samples, internal and external check samples and technical review of all results. All methods and procedures used in the lab are approved by either national or regional professional organizations. All instruments are calibrated daily and checked with high quality standards. Blank samples are routinely used to check each day's analyses. Internal check samples are used every 10 to 20 samples. The laboratory is involved in four national and international quality assurance programs. All results are double-checked for data entry accuracy and reviewed for any apparent problems.

SWFAL consists of four subdivisions: 1) soil fertility, 2) water and salinity, 3) forage and 4) animal waste. These four subdivisions use shared facilities and instruments. In addition, personnel are cross-trained between divisions. However, sample handling is different for each subdivision. Abbreviations used in this fact sheet can be found in Table 4.

Soil Fertility Tests

The soil fertility subdivision analyzes about 30,000 soil samples per year. This testing can identify nutrient status and appropriate lime and fertilizer application rates. Soil samples are received mostly from county Extension offices or crop consultants. The Extension offices also provide information on proper sampling procedures and soil bags for samples. They can also help interpret the results.

When the samples arrive at the laboratory, the information is logged into a computer system with a barcode reader, and the soils are dried at 65 C for 6 to 12 hours. The soils are then ground to pass through a 2 millimeter screen and brought into the lab for chemical analysis.

Soil pH and BI are measured by glass electrode in a 1:1 soil:water suspension and Sikora buffer solution, respectively (Sims, 1996; Sikora, 2006). Soil $\text{NO}_3\text{-N}$ and $\text{NH}_4\text{-N}$ are extracted with 1 M KCl solution and quantified by a Flow Injection Autoanalyzer (LACHAT, 1994). Plant available P, K, Ca and Mg are extracted using Mehlich 3 solution (Mehlich, 1984). Phosphorus, K, Ca and Mg in the extract are quantified by a Spectro ICP spectrometer (Soltanpour et al., 1996). Soil organic carbon and total N are determined using a dry combustion carbon/nitrogen analyzer (Nelson and Sommers, 1996). Soil sulfate is extracted by 0.008 M $\text{Ca}_3(\text{PO}_4)_2$ and analyzed by a Spectro ICP. Plant available Zn, Fe, Mn and B are extracted by DTPA-Sorbitol and quantified by ICP (Gavlak et al., 2005). Table 1 shows the brief procedures for each analysis. Soil samples normally take two to three working days to complete with reports available online.

Water and Soil Salinity Tests

The water and salinity subdivision analyzes about 10,000 samples a year. The water test identifies the suitability of water for irrigation, livestock, or rural household use (SWFAL does not analyze water for bacteria, pesticides, lead or other heavy metals; therefore, drinking water should be analyzed by a state-certified laboratory that can test these constituents). The salinity test identifies salt problems in the soil. Samples are usually submitted through the county Extension offices. These offices can supply soil bags for salinity samples and bottles for water samples. They can also provide information on good sampling procedures and help interpret laboratory results.

When the samples arrive at the laboratory, the information is logged into the computer system. Water samples are taken into the lab, filtered through Fisher P-4 paper filters, and the filtrate is analyzed. Ions in the water are either analyzed by an ICP (Standard Methods for the Examination of Water and Wastewater, 1995) or Flow Injection Autoanalyzer (LACHAT, 1994).

Soil salinity management samples are dried at a 65 C overnight and ground to pass through a 2 millimeter screen.

Table 1. Soil Fertility Procedures.

Routine Test			Secondary Test		
Analysis	Soil Amount	Procedure	Analysis	Soil Amount	Procedure
pH	10 g	Add 10 ml H ₂ O, Equilibrate half an hour, read on a pH meter.	Ca and Mg	2 g	Use P & K extract and analyze on ICP.
BI*		If pH < 6.2, add 10 ml Sikora buffer, shake one hour, read on pH meter again.	SO ₄ -S	10 g	Add 25 ml calcium mono-phosphate, shake 30 minutes, filter, analyze on ICP.
NO ₃ -N	10 g	Add 25 ml calcium sulfate solution, shake 0.5 hour, filter, analyze on flow injection analyzer using cadmium reduction chemistry.	Micronutrient Test		
P & K	2 g	Add 20 ml Mehlich 3, shake 5 minutes, filter, analyze on ICP.	Fe, Zn, and B	10 g	Add 20 ml DTPA-Sorbital solution, shake 2 hours, filter, analyze on ICP.

* BI: Buffer index for liming recommendation.

Table 2. Water and Salinity Testing Procedures.

Analysis	Offered by Tests*	Procedure
pH	5	Add 10 ml H ₂ O to 10 g soil, equilibrate 0.5 hr., read on pH meter.
pH	1, 2, 3, 6	Direct electrode reading of water samples or extract.
CO ₃ ²⁻	1, 6	Titrate with 0.02 N H ₂ SO ₄ to pH 8.3, CO ₃ ²⁻ = ml titrant x 0.02 x 6000/ ml sample.
HCO ₃ ⁻	1, 6	Titrate with 0.02 N H ₂ SO ₄ from pH 8.3 to 4.5, HCO ₃ ⁻ = ml titrant x 0.02 x 12,200/ ml sample.
EC	1, 2, 3, 5, 6	Direct electrode reading of filtered water or extract.
Na, Ca, Mg, K	1, 2, 3, 5, 6	Direct reading on ICP.
SO ₄	1, 2, 3, 6	Direct reading on ICP.
B	1, 5, 6	Direct reading on ICP.
NO ₃ -N	1, 2, 3, 6	Automated cadmium reduction.
Cl	1, 2, 3, 6	Mercuric thiocyanate flow injection analysis.
TDS	1, 2, 3, 5, 6	Greater of Σ (anions + cations) or EC x 0.66
SAR	1, 3, 5, 6	$0.043498 \times \text{Na} / [(0.04990 \times \text{Ca} + .08229 \times \text{Mg})/2]^{1/2}$
PAR	1, 3, 5, 6	$0.025577 \times \text{K} / [(0.0499 \times \text{Ca} + 0.08229 \times \text{Mg})/2]^{1/2}$
EPP	5, 6	$(10.51 \times \text{PAR} + 3.60) / [1 + (0.1051 \times \text{PAR} + 0.036)]$
ESP	5, 6	$(1.47 \times \text{SAR} - 1.26) / (0.01475 \times \text{SAR} + 0.99)$
Na %	1, 3	$0.043498 \times \text{Na} / (0.043498 \times \text{Na} + 0.08229 \times \text{Mg} + 0.04990 \times \text{Ca})$
Residual CO ₃ ²⁻	1	$(0.033328 \times \text{CO}_3^{2-} + 0.016389 \times \text{HCO}_3^-) - (0.08229 \times \text{Mg} + 0.04990 \times \text{Ca})$
Hardness	1, 2, 3	$(0.04990 \times \text{Ca} + 0.08229 \times \text{Mg}) \times 50$

* Tests: 1 = Irrigation, 2 = Livestock, 3 = Household, 5 = Salinity Management, 6 = Comprehensive Salinity

A 1:1 soil:water slurry is prepared and allowed to equilibrate for four hours, and the solution is filtered for analysis (USDA, 1954). Sodium, K, Ca, Mg, S and B in the extract are analyzed by a Spectro ICP (Soltanpour et al., 1996), and converted to the saturated paste equivalent. Electrical conductivity (EC) and pH are determined with appropriate meters. EC is also converted to the saturated paste equivalent EC. PAR, SAR, EPP and ESP are calculated using the formula from USDA (1954). Total dissolved solids (TDS) are

converted from EC and hardness is expressed as ppm of CaCO_3 (USDA, 1954). The comprehensive soil salinity samples are not dried, but prepared as a saturated paste, and then filtered for analysis similar to the salinity management samples. Table 2 illustrates the procedures to analyze and calculate the parameters offered under water and soil salinity tests. Water and salinity samples normally take three to five working days to complete with a report available online.

Table 3. Forage Testing Procedures.

Analysis	Wt (g)	Procedure
Moisture	2-3	Weigh, dry 3 hr. at 105°C, weigh after cooling.
Protein	.15-.2	Dry combustion analysis using a CN analyzer.
ADF	0.45	Weigh, digest at 100°C in ADF solution for 55 minutes, rinse 5 times in water, soak 5 minutes in acetone, dry at 105°C for 12 hours, weigh.
NDF	0.45	Use NDF solution, then same as ADF.
TDN		$88.9 - (0.779 \times \text{ADF})$.
Energy		
Maintenance		$-0.508 + (1.37 \times 0.01642 \times \text{TDN}) - [0.3042 \times (0.01642 \times \text{TDN})^2] + [0.051 \times (0.01642 \times \text{TDN})^3]$
Lactation		$(\text{TDN} \times 0.01114) - 0.054$
Gain		$-0.7484 + 1.42 \times 0.01642 \times \text{TDN} - 0.3836 \times (0.01642 \times \text{TDN})^2 + 0.0593 (0.01642 \times \text{TDN})^3$
RFV		$[88.9 - (0.779 \times \text{ADF})] \times (120/\text{NDF}) \times 0.775$
Minerals	0.5	Wet digestion and ICP

Table 4. Common Abbreviations.

Abbreviation	Definition
BI	Buffer index
ICP	Inductively coupled plasma spectrometer
EC	Electrical conductivity
TDS	Total dissolved solids
SAR	Sodium absorption ratio
PAR	Potassium absorption ration
EPP	Exchangeable potassium percent
ESP	Exchangeable sodium percent
ADF	Acid detergent fiber
NDF	Neutral detergent fiber
TDN	Total digestible nutrients
RFV	Relative feed value

Forage Analysis

The forage subdivision analyzes more than 10,000 plant samples a year to determine forage quality and nitrate levels. Forage quality is determined by measuring protein, ADF, NDF, moisture and minerals. The turn-around time varies with the tests desired. However, all work is normally completed in three to five working days. Nitrate testing is important to avoid feeding poisonous forage to livestock. Since this information is vital, nitrate results are posted on the website as soon as results are obtained from the laboratory. Nitrates are normally analyzed in one to two working days.

After logging in the forage sample, it is prepared for analysis by weighing the sample, drying the sample at 85 C for about 12 hours (very wet samples may take longer), weighing the sample after drying for moisture calculation, grinding to pass through a 1.0 millimeter screen, and taking the sample to the lab for analysis. Procedures and calculations are shown in Table 3.

The final moisture content of plant samples is determined by drying ground sample at 105 C overnight. Total nitrogen (TN) and carbon are determined using a dry combustion Carbon/Nitrogen Analyzer (NFTA, 1993) and crude protein is calculated by multiply TN by 6.25 for all samples except for wheat grain which has a conversion factor of 5.7. Acid detergent fiber, neutral detergent fiber and acid detergent lignin are determined using the Ankom Fiber Analyzer (Ankom Technology, Macedon, NY, 2011). Mineral contents of the forage are analyzed by a Spectro ICP following acid digestion (NFTA, 1993).

Animal Waste Analysis

The animal waste subdivision analyzes about 2,000 samples per year. Nutrients in animal wastes samples are extracted or digested following procedures in the Recommended Methods for Manure Analysis (2003). Total N is determined by a dry combustion CN Analyzer. Sodium, Ca, Mg, K, S and P in the extracts or digests are quantified by a Spectro ICP using the Standard Methods (1995) after wet digestion.

References

- ANKOM Technology. 2011. Method for determining acid detergent fiber. <https://www.ankom.com/procedures.aspx>
- Gavlak, R., D. Horneck, and R.O. Miller. 2005. Soil, plant and water reference methods for the Western Regions. 3rd ed. Oregon State University.
- LACHAT. 1994. QuickChem Method 12-107-04-1-B. LACHAT Instrument, Milwaukee, WI.
- Mehlich, A. 1984. Mehlich 3 soil test extractant: A modification of Mehlich 2 extractant. *Commun. Soil Sci. Plant Anal.* 15:1409-1416.
- National Forage Testing Association. 1993. Forage Analyses Procedures. http://www.foragetesting.org/index.php?page=lab_procedures
- Nelson and Sommers. 1996. Total carbon, organic carbon, and organic matter. In D.L. Sparks (ed.) *Methods of Soil Analysis. Part 3. Chemical Methods.* SSSA Book Ser: 5. SSSA and ASA, Madison, WI.
- Recommended Methods for Manure Analysis. 2003. University of Wisconsin-Extension. <http://learningstore.uwex.edu/assets/pdfs/A3769.PDF>
- Sikora, F.J. 2006. A buffer that mimics the SMP buffer for determining lime requirement of soil. *Soil Sci. Soc. Am. J.* 70: 474-486.
- Sims, J.T. 1996. Lime requirement, pp. 491-515. In: D.L. Sparks (ed.) *Methods of Soil Analysis, Part 3. Chemical Methods.* SSSA Book Ser: 5. SSSA and ASA, Madison, WI.
- Soltanpour, P.N., G.W. Johnson, S.M Workman, J. B. Jones, Jr., and R.O. Miller. 1996. Inductively coupled plasma emission spectrometry and inductively coupled plasma-mass spectrometry. Pp. 91-139. In: D.L. Sparks (ed.) *Methods of Soil Analysis, Part 3. Chemical Methods.* SSSA Book Ser: 5. SSSA and ASA, Madison, WI.
- Standard Methods for the Examination of Water and Wastewater. 1995. 19th Edition.
- USDA Salinity Laboratory. 1954. Diagnosis and improvement of saline and alkali soils. *Agriculture Handbook No. 60.* Riverside, CA.

Oklahoma State University, in compliance with Title VI and VII of the Civil Rights Act of 1964, Executive Order 11246 as amended, and Title IX of the Education Amendments of 1972 (Higher Education Act), the Americans with Disabilities Act of 1990, and other federal and state laws and regulations, does not discriminate on the basis of race, color, national origin, genetic information, sex, age, sexual orientation, gender identity, religion, disability, or status as a veteran, in any of its policies, practices or procedures. This provision includes, but is not limited to admissions, employment, financial aid, and educational services. The Director of Equal Opportunity, 408 Whitehurst, OSU, Stillwater, OK 74078-1035; Phone 405-744-5371; email: eeo@okstate.edu has been designated to handle inquiries regarding non-discrimination policies; Director of Equal Opportunity. Any person (student, faculty, or staff) who believes that discriminatory practices have been engaged in based on gender may discuss his or her concerns and file informal or formal complaints of possible violations of Title IX with OSU's Title IX Coordinator 405-744-9154.

Issued in furtherance of Cooperative Extension work, acts of May 8 and June 30, 1914, in cooperation with the U.S. Department of Agriculture, Director of Oklahoma Cooperative Extension Service, Oklahoma State University, Stillwater, Oklahoma. This publication is printed and issued by Oklahoma State University as authorized by the Vice President for Agricultural Programs and has been prepared and distributed at a cost of 20 cents per copy. Revised 0518. GH.