Laboratory Services Division Bureau of Soils and Water Management

TM-LSD-04-04

Department of Agriculture		
SECTION : SOIL CHEMISTRY	Issue No.: 3	Effective date: May 27, 2024
SUBJECT : TOTAL NITROGEN	Revision No.: 2	Page 1 of 8

SCOPE

This procedure shall be applied when total nitrogen or organic nitrogen needs to be determined on soil samples.

PRINCIPLE

Nitrogen is an essential element required for the plant growth. Organic nitrogen may consist 97-98 percent of the total N in the soil. Inorganic nitrogen compounds such as NH_4^+ , NO_2^- , and NO_3^- may consist of 2-3%, these are the forms of Nitrogen that are absorbed by the plants. Nitrogen increases protein content of plants directly. Enough N produces a dark green color in the leaves, caused by a high concentration of chlorophyll. N deficiency causes Chlorosis (a yellowing of the leaves from declining chlorophyll).

There are some major forms of N occur in the soil such as organic N that part of soil organic matter that unavailable to growing plant, ammonium N fixed by clay minerals that very slowly available to plants and ammonium and nitrate ions or soluble compounds that the N which plant use. Nitrates are highly mobile in soil. They move freely with soil water. So much nitrate-N may leach through the soil profile-more deep, sandy soils than on fine textured soils with moderate drainage and high rainfall.

Nitrogen fertilizer affects the soil acidity when the nitrification process converts the ammonium ion to nitrate, hydrogen ions are released. Nitrogen fertilizers containing or forming ammonium-N increases soil acidity unless the plant absorbs the ammonium ion directly. Also, nitrate is a major factor associated with leaching of such bases as calcium, magnesium, and potassium from the soil. The nitrate and bases move out together. These bases are removed and replaced by hydrogen, soils become more acidic. The excess nitrogen is lost in ionic or gaseous form through leaching, volatilization, and denitrification, if nitrate is not absorbed by plant roots, it is carried away by runoff or leaches into the soil along with water. The phytoavailability of the nitrogen pool increases when excess nitrogen is applied, and this increase intensifies the potential threat to the surrounding environment. There are close relationships between the excessive application of nitrogen fertilizers and environmental problems such as eutrophication, the greenhouse effect, and acid rain. Consuming contaminated groundwater or crops with a high concentration of nitrate has negative effects on human health.

The Kjeldahl procedure for the determination of total N involves the digestion of sample to convert organic N to NH_4^+ -N and determination of the NH_4^+ -N in the digest. Soil organic matter is oxidized by heating the sample with boiling concentrated H_2SO_4 containing potassium sulfate-catalyst mixture converting organic-N into ammonium sulfate and promoting oxidation. With the salt mixture, K_2SO_4 indicator increases the temperature of digestion allowing a more effective oxidation; while copper increases the rate of oxidation of organic matter by H_2SO_4 .

The NH₄⁺-N in the digest is determined by collecting the NH₃-N liberated by distillation of the digest with NaOH into dilute boric acid indicator. The boric acid captures the ammonia gas, forming an ammonium-borate complex. As the ammonia collects, the color of the receiving solution changes. The addition of sulfuric acid during titration exactly neutralizes the ammonium borate complex, and reverse color change is produced.

This procedure is satisfactory for total N analysis of most nitrogenous compounds in soil samples. If the soil for analysis contains significant amounts of inorganic-N in the form of NO_3^- and NO_2^- , this N is not recovered quantitatively by this procedure

Prepared by:	Reviewed by:	Approved by:	
embyamboa EZRA MAE B. GAMBOA	JP Sanchy FLORFINA P. SANCHEZ	GINA P.NILO. Ph.D.	
Document Controller	Head, Soil Chemistry Section	Quality Manager	
THIS IS A CONTROLLED DOCUMENT MAINTAINED ELECTRONICALLY.			
THIS IS UNCONTROLLED WHEN DOWNLOADED, PRINTED AND PHOTOCOPIED.			

TEST METHODS N Laboratory Services Division Bureau of Soils and Water N Department of Agriculture	MANUAL Management	TM-LSD-04-04		
SECTION : SOIL CHEMISTRY		Issue No.: 3	Effective date: May 27, 2024	
SUBJECT : TOTAL NITROGEN		Revision No.: 2	Page 2 of 8	
1. Degradation of Sample (Digestion) Nitrogenous compounds in soil sample+H ₂ SO ₄ K_2SO_4 (NH ₄) ₂ SO ₄ (aq) + CO ₂ (g) + SO ₂ (g) + H ₂ O(g)				
$CuSO_4$ For quantitative determination of NO ₃ -N and NO ₂ -N:				
Na ₂ S ₂ O ₃ Nitrate + Salicylic acid + H2SO4> Nitro-salicylic acid amino salicylic acid				
Amino salicylic acid + H_2SO_4 K_2SO_4 (NH ₄) ₂ SO ₄ + CO ₂ + SO ₂ + H_2O CuSO ₄				
2. Liberation of ammonia (Distillation)				
(NH ₄) ₂ SO ₄ (aq) + 2NaOH> Na ₂ SO ₄ (aq) + 2H ₂ O(I) + 2NH ₃ (g)				
3. Capture of ammonia (Distillation	n)			
$H_3BO_3 + H_2O + NH_3$ > NH_4 + : $H_2BO_3^-$ + H_3BO_3 ammonium borate complex excess boric acid				
4. Nitrogen Determination (Direct	Titration)			
2NH.	4H2BO3 - + H2SO4 H2BO3 ⁻ + H ⁺	> (NH4)2SO4 + 2H > H3BO3	l₃BO₃	
TEST PRECAUTIONS The use of high concentration of K_2SO_4 speeds up the period of digestion and therefore prevents bumping and spattering also high concentration of K_2SO_4 causes the digest to solidify upon cooling, which means that considerable time may be required to take up the digest with water before distillation. Another disadvantage is serious frothing during digestion of organic soils.				
The addition of distilled water after cooling the digested sample is to prevent the complete adherence of salt mixture on the glass wall of the digestion tube.				
Perform distillation cleaning using distilled water before and after the analysis.				
Work on the fume hood when dispensing concentrated sulfuric acid to the digestion tubes with soil and salt mixture. Cool the digests before adding water, hot concentrated acid violently reacts with water that will cause the digests to shoot out of the digestion tube.				
Prepared by: embgamboa	Reviewed by: JP Aanck	× '	Approved by:	
EZRA MÁE B. GAMBOA	FLORFINA P. SANCH	ez o	SINĂ PUNILO, Ph.D.	
Document Controller	Head, Soil Chemistry	Section	Quality Manager	

THIS IS A CONTROLLED DOCUMENT MAINTAINED ELECTRONICALLY. THIS IS UNCONTROLLED WHEN DOWNLOADED, PRINTED AND PHOTOCOPIED.

TEST METHODS MANUAL

Laboratory Services Division

Department of Agriculture

TM-LSD-04-04

Work on the fume hood during the preparation of 10M sodium hydroxide, fumes evolve when dissolving the base. Slowly add the water to base to prevent splashing. Stir continuously to prevent crystallization of sodium hydroxide.

Always verify the volume of NaOH dispensed by the equipment prior to use.

Always check the tubing and piston of the digital burette if there are formation of gas bubbles.

EQUIPMENT

- a) Block digester
- b) Steam distillation apparatus
- c) Balance, precision of 0.0001 g
- d) Digital Titrator
- e) Dispenser (1-10 ml calibration

Limited Preview

For full access, kindly fill out the Document Request

- b) Digestion tube stand or rack, 50 holes Form.
- Google Form link:
- e) Burettes 50 mL (if Digital titrator is not available)
- f) Volumetric pipette, (https://forms.gle/RbCgCdA54prTS6oN7
- g) Graduated pipettes (10, 25 mL)
- h) Graduated cylinder (50 ml)
- i) Erlenmeyer flasks, 125 mL
- j) Reagent bottles
- k) Funnels, 70 mm.
- l) Volumetric flasks (1L, 2L, 5L)
- m) Pipettor

Thank you!

CHEMICALS AND REAGENTS

- a) Potassium sulfate and Copper Sulfate- salt catalyst mixture: Prepare a mixture of 500 g of K₂SO₄ (90% of the salt mixture) and 55.56 g of CuSO₄.5H₂O (10% of the salt mixture). Pulverize the reagents separately before mixing, and grind the mixture using mortar and pestle to powder the cake that forms during mixing.
- b) Sulfuric acid (H₂SO₄), concentrated (18 *M*)
- c) Sodium hydroxide (NaOH) solution, approximately 10 *M*. Place 2.0 kg of reagent-grade NaOH in a heavy-walled 5-L Pyrex bottle. Add 3L of distilled water, and swirl the flask until the alkali is dissolved. Cool the solution before filling the flask up to mark with distilled water Then swirl the bottle vigorously to mix the contents, and fit the neck with some arrangement that permits the alkali to be stored and dispensed with protection from atmospheric CO₂.
- d) Mixed Indicator Solution: Dissolve 0.099g of bromocresol green and 0.066g of methyl red using 95% ethanol in a 100 mL volumetric flask, fill up to mark.
- e) Boric acid-indicator solution: Place 40 g of pure boric acid in a 2-L beaker. Add about 1800 ml of distilled water, heat and swirl until the H₃BO₃ is dissolved. (Dissolution of the H₃BO₃ also can be affected by vigorous stirring with

THIS IS A CONTROLLED DOCUMENT MAINTAINED ELECTRONICALLY. THIS IS UNCONTROLLED WHEN DOWNLOADED, PRINTED AND PHOTOCOPIED.