

Compost Analysis Procedures

DRY ASHING PROCEDURE FOR COMPOSTS

Reagents and Apparatus

1. Hydrochloric Acid, 6N HCl.
2. Muffle furnace.
3. 15 ml crucibles.
4. Kimax glass tubes with 50 ml graduation mark.
5. Dilutor.
6. Magnesium nitrate solution- Add 113 g of magnesium oxide to 300 ml of water and stir to a paste. Add 7.5N nitric acid until the magnesium oxide is in solution. Add a little excess magnesium oxide and boil. Filter and dilute to 1 liter.

Procedure

1. Weigh out 0.500g of ground and dried (65°C) compost material into each 10 ml crucible.
2. Place the crucibles in a cool muffle furnace. Set furnace temperature to reach 500°C (about 2-3 hours).
3. After 5 hours of muffling at 500°C, turn off the furnace and let it cool. Do not open the door of the furnace during the ashing and cooling.
4. Add 10 ml of 6N HCl into each crucible to dissolve the ash.
5. Transfer all of the stuff in the crucible to a Kimax glass tube, and dilute to 50 ml mark with distilled water, then mix it thoroughly.
6. Allow suspended materials to settle to the bottom of the tube, or filter with Whatman #42 filter paper.
7. The filtered solution is ready to determine K, B, Ca, Mg, Zn, Fe, Mn, and Cu, with or without further dilution.
8. The sulfur in the plant tissues should be oxidized to sulfate by heating with magnesium nitrate first then do dry ashing.
 - A. Repeat step 1 above.
 - B. Moisten the plant material in the crucible with 10 ml of magnesium nitrate solution
 - C. Place the crucible on a hot plate and evaporate until the action is complete.
 - D. Transfer crucible while hot to a muffle furnace.
 - E. Repeat steps 3, 4, 5, 6, and 7.

References

1. Chapman H. D. and P. F. Pratt. 1961. *Methods of Analysis for Soils, Plants, and Waters*. University of California, Riverside, CA.
2. Jones, Jr., J. B. and V. W. Case 1990. Sampling, Handling, and Analyzing Plant Tissue Samples (Chapter 15) pp389-427. *In: Soil Testing and Plant Analysis*, 3rd ed. -SSSA book series, no. 3. 677 S. Segon Rd., Madison, WI.
3. Jones, Jr., J. B., B. Wolf, and H. A. Mills 1991. Preparation and Analysis (Chapter 4) pp23-26. *In: Plant Analysis Handbook. a practical sampling, preparation, analysis, and interpretation guide*. Micro-Macro Publishing, Inc. 183 Paradise Blvd., Suite 108, Athens, Georgia.

SATURATED PASTE FOR GREENHOUSE MEDIA AND COMPOSTS

Equipment

1. Vacuum pump
2. Suction manifold: A suitable apparatus may be constructed from normal lab equipment. A set of suction flasks can be used as collection flasks.
3. Buchner funnels can be inserted into rubber stoppers and placed on top of the suction flasks.
4. Whatman No. 2 filter paper: 11 cm
5. 600-ml beakers.

Reagents

1. 0.01 N KCl
Dissolve 0.746 g of KCl (oven dried 105°C) in distilled water and dilute to 1 liter (E. C. = 141.2 x 10⁻⁵ mhos/cm at 25°C).

Procedure

1. Transfer undried compost or greenhouse media into 600-ml beakers. Beaker should be 1/2 to 3/4 full.
2. Add distilled water to beaker until soil saturated. Mix constantly with spatula. At saturation, the soil paste glistens as it reflects light. The soil will flow slightly when the container is tipped and the paste slides freely and cleanly off the spatula. Free water should not collect on the surface.
3. Determine pH of the soil paste after 30 minutes using pH meter by carefully inserting the electrodes (wiggle the electrodes gently to attain good solution contact).
4. Allow paste to stand for an additional 30 minutes before filtering. Filter solution from paste using suction and manifold 1 to 4 hours after water is first added to the soil.
5. Transfer filtrate from suction flask to the test tubes.
6. Determine nitrate-nitrogen of the filtered extract by a Flow Injection Analyzer based on the Lachat Method No 12-107-04-1-B.
7. Determine electrical conductivity of the filtered extract by E. C. bridge directly.
8. Determine phosphorus of the filtered extract by colorimeter at 660 nm.
9. Determine Potassium, Calcium and Magnesium of the filtered extract by an atomic absorption/emission spectrometer.

Calibration and Standards

1. 1000 ppm P, K, Ca and Mg stock solution
2. Working standards
Pipette the following volumes of 1000 ppm stock solution into 1000 ml volumetric flasks and dilute to volume with D.I water.

mls of 1000 ppm stock				Final working Standards			
K	Na	Ca	Mg	K	Na	Ca	Mg
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
20.0	15.0	50	5.0	20.0	15.0	50	5.0
40.0	30.0	100	10.0	40.0	30.0	100	10.0

Store in plastic bottles and keep in the refrigerator until ready to use.

References

1. Warncke, D. 1998. Greenhouse root media. pp. 61-64. *In* J. R. Brown (ed.) recommended chemical soil test procedures for the North Central Region. NCR Publication No. 221 (Revised). Missouri Agricultural Experiment Station SB 1001.
2. Greenhouse soil test 1987. P. 24-26. *In* E. E. Schulte, J. B. Peters and P. R. Hodgson edited. Wisconsin Procedures for Soil Testing, Plant Analysis and Feed and Forage Analysis. University of Wisconsin-Extension, Madison, WI.
3. Whipker, Brian E., Terri Kirk and P. Allen Hammer. 1994. Industry root media analysis results: Useful in determining greenhouse nutrition problems and educational opportunities. *Comm. Soil Sci. Plant anal.* 25:1455-1460.

KJELDAHL NITROGEN AND PHOSPHOROUS IN COMPOSTS

Reagents and Apparatus

1. Concentrated Sulfuric Acid (H₂SO₄) reagent grade.
2. Digestion tablets (Kjeltab).
3. Glass boiling beads.
4. Aluminum digestion block with temperature controller.
5. Kimax glass tubes with 50-ml graduation mark.

Procedure

1. Weigh out 0.300g of compost sample into each 50 ml digestion tube.
2. Add one digestion tablet (Kjeltab) and two glass boiling beads to each tube.
3. Under hood, add 3.5 ml of concentrated sulfuric acid (H₂SO₄) using an acid resistant 5-ml repipet device.
4. Include at least one Blank and one Check each batch.
5. Preheat aluminum digester to 180 °C. This takes a half hour to reach temperature.
6. Place tubes in block digester which has been preheated and continue to heat to 390 +/-5°C. Then digest them for two hours.
7. Remove the tubes from the heating block and allow about 10 minutes for cooling.
8. Add 10 - 15 ml of distilled water to the digestion tubes while they are still warm. Mix them to dissolve any crystals that may have formed.
9. Dilute to the 50-ml mark with distilled water and mix thoroughly after capping.
10. Analyze ammonium for TKN on a Flow Injection Analyzer based on the Lachat Method No 13-107-06-2-D.
11. Analyze orthophosphate for TKP on a Flow Injection Analyzer based on the Lachat Method No 13-115-01-1-B.

Calculations

To get compost TKN %, $N \% = \text{reading (mg/l)} \times 50 / 0.300 / 10000$

To get compost TKP %, $P \% = \text{reading (mg/l)} \times 50 / 0.300 / 10000$

References

1. QuikChem Automated Ion Analyzer Methods Manual. No. 13-107-06-2-D, December, 1996. Determination of total Kjeldahl nitrogen in soils and plants by flow injection analysis (Block Digester Method), LACHAT Instruments, Milwaukee, WI.
2. AOAC Official Methods of Analysis 1990. Protein (Crude) in animal feed, semiautomated method No. 976.06, p72.
3. Jones, Jr., J. B., B. Wolf, and H. A. Mills 1991. Methods of Elemental Analysis (Chapter 4) pp27-38. *In*: Plant Analysis Handbook. Micro-Macro Publishing, Inc. 183 Paradise Blvd., Suite 108, Athens, Georgia.
4. QuikChem Automated Ion Analyzer Methods Manual. No. 13-115-01-1-B, December, 1996. Determination of phosphorous in soils and plants by flow injection analysis (Block Digester Method). LACHAT Instruments, Milwaukee, WI.

TOTAL K, NA, Ca AND Mg TESTS FOR COMPOSTS

Equipment

1. Adjustable pipette.
2. 20-ml glass beaker.
3. Atomic Absorption Spectrophotometer.

Reagents

1. 1.2 N HCl solution
Add 103.7 ml of hydrochloric acid (sp. Gr. 1.19, 37.5%) into 1 liter bottle. Dilute with distilled water and mix well.
2. 0.105 % La diluent
Place 1.2314 g lanthanum oxide (La_2O_3), low calcium grade, in a one-liter volumetric flask. Add 4 ml of 6 N HCl to dissolve the La_2O_3 and then dilute to one liter with demineralized water.

Procedure

1. Transfer filtrate from the dry ashing procedure into 10-ml tubes.
2. Dilute 1.0 ml of the filtrate with 9.0 ml of the 1.2 N HCl solution. This is a 10 times diluted filtrate.
3. Dilute 0.5 ml of the diluted filtrate with 9.5 ml of the 0.105% La diluent.
4. Read samples on atomic absorption spectrophotometer using appropriate standards and instrument settings for Ca and Mg.
5. Flame emission spectrometers may be used for determination of K and Na directly in the diluted filtrate.
6. This diluted filtrate is also used for determination of P.

Calibration and Standards

1. 1000 ppm K, Na, Ca and Mg stock solution
2. Working standards
Pipette the following volumes of 1000 ppm stock solution into 1000 ml volumetric flasks and dilute to volume with 1.2 N HCl solution.

mls of 1000 ppm stock				Final working Standards			
K	Na	Ca	Mg	K	Na	Ca	Mg
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
20.0	15.0	50	5.0	20.0	15.0	50	5.0
40.0	30.0	100	10.0	40.0	30.0	100	10.0

Store in plastic bottles and keep in the refrigerator until ready to use.

Calculations

Ca and Mg % in plant = ppm in reading x 10 x 50 / 0.500 / 10000

K and Na % in plant = ppm in reading x 10 x 50 / 0.500 / 10000

References

1. Warncke, D., and J. R. Brown 1998. Potassium and other basic cations. pp. 31-34. *In* J. R. Brown (ed.) recommended chemical soil test procedures for the North Central Region. NCR Publication No. 221 (Revised). Missouri Agricultural Experiment Station SB 1001.
2. Thomas, G. W. 1982. Exchangeable cations. P. 159-165. *In* A. L. Page et al. (ed). Methods of soil analysis. Part 2. 2nd ed. Agron. Monogr. 9. ASA and SSSA, Madison, WI.
3. Brown, J. R., and R. R. Rodriguez. 1983. Soil testing. A guide for conducting soil tests in Missouri. EC923. Extension Division, University of Missouri-Columbia.

MICRONUTRIENT TESTS FOR COMPOSTS

Equipment

1. Adjustable pipette.
2. 20-ml glass beaker.
3. Atomic Absorption Spectrophotometer.

Reagents

1. 1.2 N HCl solution
Add 103.7 ml of hydrochloric acid (sp. Gr. 1.19, 37.5%) into 1 liter bottle. Dilute with Distilled water and mix well.

Procedure

1. Transfer filtrate from the dry ashing procedure into 10-ml tubes.
2. Read samples on atomic absorption spectrophotometer using appropriate standards and instrument settings. Set zero with reagent blank, which is 1.2 N HCl solution.
3. Report as ppm Zn, Fe, Mn or Cu in plant.

Calibration and Standards

1. 1000 ppm Zn, Fe, Mn, and Cu stock solution
2. 100 ppm Zn, Fe, Mn, and Cu working stock solution
Dilute 10 ml of 1000 ppm stock solution each to 100 ml with deionized water, respectively.
3. Working standards
Pipette the following volumes of 100 ppm working stock solution into 400 ml volumetric flasks and dilute to volume with 1.2 N HCl solution:

ml of 100 ppm stock				Final working Standards			
Zn	Fe	Mn	Cu	Zn	Fe	Mn	Cu
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2.00	12.0 0	4.00	1.00	0.50	3.00	1.00	0.25
4.00	24.0 0	8.00	2.00	1.00	6.00	2.00	0.50

Store in plastic bottles and keep in the refrigerator until ready to use.

Calculations

Micronutrient ppm in plant = ppm in reading x 100

References

1. Whitney, D. A. 1998. Micronutrients: Zinc, Iron, Manganese and Copper. pp. 41-44. *In* J. R. Brown (ed.) recommended chemical soil test procedures for the North Central Region. NCR Publication No. 221 (Revised). Missouri Agricultural Experiment Station SB 1001.
2. Cox, F. P., and E. J. Kamprath. 1972. Micronutrient soil tests. P. 289-317. *In* J. J. Mortvedt et al. (ed). Micronutrients in Agriculture, Soil Sci. Soc. Amer. Inc., Madison, Wis.
3. Brown, J. R., and R. R. Rodriguez. 1983. Soil testing. A guide for conducting soil tests in Missouri. EC923. Extension Division, University of Missouri-Columbia.
4. Lindsay, W. L., and W. A. Norvell. 1978. Development of a DTPA soil test for zinc, iron, manganese, and copper. *Soil Sci. Soc. Amer. J.* 42:421-428.
5. Chapman, H. D., and P. F. Pratt. 1961. Methods of analysis for soils, plants and waters. University of California, Riverside, CA.