

Total Phosphorus in Organic Fertilizer

1. Scope:

To provide a standardized procedure for the gravimetric analysis of total phosphorus in organic fertilizer using the quinolinium molybdophosphate method.

2. Principle:

Samples are ashed with magnesium nitrate, dissolved in *aqua regia*, and precipitated with Quimociac reagent to determine the amount of total phosphorus present. Samples are prepared according to Sample Preparation, Storage, and Disposal (RA-SP-SMPL-PREP).

3. Safety:

- 3.1. All laboratory safety rules for chemical handling, sample preparation, and analysis shall be followed. Read the SDS for all materials before use.
- 3.2. Nitric acid and hydrochloric acid are highly corrosive. Preparation of the Quimociac reagent and *aqua regia* shall be done in a fume hood using appropriate personal protective equipment (gloves, eye protection, etc.)

4. Definitions:

QMP = quinolinium molybdophosphate = $(C_9H_7N)_3H_3PO_4 \cdot 12MoO_3$

5. Equipment (equivalents are acceptable):

- 5.1. Analytical balance capable of weighing to 0.0001g
- 5.2. Oven capable of $250^{\circ}C \pm 25^{\circ}C$
- 5.3. Muffle furnace capable of $600^{\circ}C \pm 50^{\circ}C$
- 5.4. Hot plate
- 5.5. Vycor beaker
- 5.6. Volumetric flat bottom boiling flask – 250mL
- 5.7. Erlenmeyer flask – 250mL
- 5.8. Vacuum filter flask with adapter – 2L
- 5.9. Gooch crucibles
- 5.10. Glass fiber filter – 2.4cm circles (Whatman 934-AH)
- 5.11. Glass fiber filter – 11cm circles (Whatman 934-AH)
- 5.12. Boiling chips (micro granules)
- 5.13. Desiccator

6. Reagents and Supplies (equivalents are acceptable):

- 6.1. Nitric acid, concentrated (Fisher cat# A509-P212)
- 6.2. Hydrochloric acid, concentrated (Fisher cat# A144C-212 or A508-4)
- 6.3. Ethanol (Pharmco cat# 111000190 or 111000200)
- 6.4. Sodium molybdate dihydrate (Fisher cat# S336-3)
- 6.5. Citric acid (VWR cat# BDH9228)
- 6.6. Synthetic quinoline (Acros organics cat# 221141000 or Sigma Aldrich cat# 241571)
- 6.7. Acetone (Fisher cat# A949-4)
- 6.8. Magnesium nitrate hexahydrate (Fisher cat# M464-500)

7. Preparation of Reagents:

- 7.1. Prepare the *aqua regia* by mixing 400mL water, 1200mL concentrated hydrochloric acid, and 400mL concentrated nitric acid. Allow to vent in a fume hood.
- 7.2. Prepare the Quimociac reagent:
 - 7.2.1. Dissolve 70g sodium molybdate dihydrate in 150mL water.
 - 7.2.2. In a 1L volumetric flask, dissolve 60g citric acid in a mixture of 85mL concentrated nitric acid and 150mL water. Allow to cool.
 - 7.2.3. Gradually add the sodium molybdate solution to the citric acid solution while stirring.
 - 7.2.4. Dissolve 5mL synthetic quinoline in a mixture of 35mL concentrated nitric acid and 100mL water.
 - 7.2.5. Gradually add the quinoline solution to the molybdate-citric acid solution. Mix and let stand for 24 hours.
 - 7.2.6. Filter through an 11cm glass fiber filter.
 - 7.2.7. Add 280mL acetone and fill to the mark with water.
- 7.3. Prepare the magnesium nitrate solution by dissolving 500g $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 525mL DI water.

8. Analysis

- 8.1. Perform the daily balance verification.
- 8.2. Weigh ~1g sample into a Vycor beaker. Record weight to nearest 0.0001g.
- 8.3. Moisten dry fertilizer samples with ethanol (liquid samples do not require ethanol).
- 8.4. Add 5mL of the magnesium nitrate solution.
- 8.5. Place on a hot plate and evaporate to dryness.

- 8.6. Place a watch glass on the beaker and place in a 600°C furnace for ~2 hours. Allow to cool.
- 8.7. Add 25mL of water, 15mL *aqua regia*, and swirl to mix well.
- 8.8. Heat on a hot plate with watch glass at 200°C for 40 minutes.
- 8.9. Rinse the watch glass into the beaker and fill the beaker ~halfway with DI water.
- 8.10. Using a funnel, pour the contents of the beaker into a 250mL boiling flask. Rinse the beaker with DI water 2-3 times and add to the flask. Cool to room temperature then fill to the mark with DI water and mix thoroughly.
- 8.11. Allow the particulates to settle overnight.
- 8.12. Pipette into a 500mL Erlenmeyer flask a suitable aliquot of the clear supernatant to form ~0.3g precipitate. If the guarantee is $\leq 5\%$, aliquot 50mL. If the guarantee is 5-10%, aliquot 25mL. If the guarantee is $>10\%$, aliquot 15mL.
- 8.13. Add water to bring the total volume to ~100mL.
- 8.14. Heat the solution for 15 minutes on a hot plate preheated to ~350°C.
- 8.15. Remove from the heat, swirl, and add 50mL Quimociac reagent. Swirl again then resume heating until it boils again. Boil for 1 minute. Set aside and cool to room temperature, swirling 3-4 times while it is cooling.
- 8.16. Weigh a Gooch crucible fitted with a glass fiber filter. Record the weight to the nearest 0.0001g.
- 8.17. Using the vacuum flask and vacuum, filter the precipitate into the crucible.
- 8.18. Wash the precipitate with five 5mL portions of water, allowing each portion to drain thoroughly before adding the next.
- 8.19. Dry the crucible for 45 minutes in an oven preheated to 250°C.
- 8.20. Cool in a desiccator to room temperature.
- 8.21. Weigh the crucible and record weight to nearest 0.0001g. Subtract the weight of the crucible and filter from step 8.16 to determine the weight of the precipitate.
- 8.22. If the weight of the precipitate is greater than 1.0g, repeat steps 8.12 – 8.21 using a smaller aliquot of clear supernatant.

9. QA/QC:

9.1. A laboratory control sample (LCS) shall be run with each set. An acceptable LCS is a AAFCO or Magruder check sample with the reported mean and standard deviation for the total phosphorus (gravimetric method). An acceptable recovery is ± 2 standard deviations.

9.2. The reporting limit (RL) is 0.05%.

10. Calculations:

Calculate percent total phosphoric acid (P_2O_5):

$$\% P_2O_5 = \frac{W * D * 0.03207 * 100}{S}$$

Where:

W = Weight (g) of precipitate from step 8.21

D = Dilution factor = 250mL/aliquot

S = Sample weight (g)

0.03207 = Gravimetric factor derived from

Molecular weight of P_2O_5 = 141.94

Molecular weight of QMP = 2212.71

$$\frac{P_2O_5}{2QMP} = \frac{141.94}{2 * 2212.71} = 0.03207$$

11. References:

AOAC International Official Methods of Analysis, Method 962.03 (chapter 2.3.07), 17th edition, 2000.

Preparation of Quimociac Reagent, AOAC International Official Methods of Analysis, Method 962.02A (b) and (c) (chapter 2.3.03), 17th edition, 2000.

USDA Food Safety and Inspection Service, Chemistry Laboratory Guidebook, Method 3.009, June 1987.

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