

## Total Phosphorus in Fertilizer

### 1. Scope:

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

### 2. Principle:

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

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

## 7. Preparation of Reagents:

- 7.1. Prepare the *aqua regia* by mixing 400mL DI 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 DI water.
  - 7.2.2. In a 1L volumetric flask, dissolve 60g citric acid in a mixture of 85mL concentrated nitric acid and 150mL DI 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 DI 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 DI 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. Verify the balance to be used.
- 8.2. Weigh ~1g sample into a Vycor beaker (weigh ~0.5g if guarantee is  $\geq 40\%$ ). 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 prepared in 7.3.

- 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 DI 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 a suitable aliquot of the clear supernatant to form ~0.3g precipitate into a 500mL Erlenmeyer flask.

| <b>Guarantee</b> | <b>Suggested Amount</b> |
|------------------|-------------------------|
| <5%              | 50mL                    |
| 5 – 10%          | 25mL                    |
| >10%             | 15mL                    |

- 8.13. Add DI 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 DI 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 (~1 hour).

- 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  $\pm 2$  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), 17<sup>th</sup> edition, 2000.

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

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



