Available Phosphorus (P_2O_5) in Solid Inorganic Fertilizer
Direct Extraction Method

1. Scope:

To provide a standardized procedure for the analysis of available inorganic phosphorus (as P_2O_5) in solid fertilizer and bat guano.

2. Principle:

Samples are prepared according to *Fertilizer Sample Preparation, Storage, and Disposal (FEED/FERT SP-3).* The sample is extracted with ammonium citrate at pH 7.0 in the presence of disodium EDTA (ethylenedinitrilotetraacetic acid) to complex the calcium and magnesium. The available phosphorus is precipitated with Quimociac Reagent, and the resulting precipitate is filtered, washed, dried, and weighed to calculate the amount of available phosphorus (as P_2O_5) present in the sample.

3. Safety:

3.1 Consult the appropriate SDS on the safe handling of all chemicals used in this method.

3.2 Nitric acid is highly corrosive. Preparation of the Quimociac reagent and the nitric acid (1+1) should be done in a fume hood using the appropriate protective clothing, eye protection, and gloves.

3.3 Aqueous ammonium hydroxide is toxic and produces vapors that are very irritating to the skin and respiratory tract. Preparation of the ammonium citrate/EDTA solution shall be done in a fume hood using the appropriate protective clothing, eye protection, and gloves.

4. Apparatus and Equipment:

4.1. Analytical balance (Mettler Toledo XS204 or equivalent)

4.2. Laboratory oven (Fisher Scientific ISOTEMP Oven 725F or equivalent)

4.3. Water bath with shaker (capable of maintaining temperature of 65°C ± 3°C)

4.4 Hot plate (Thermo Scientific HPA2235MQ or equivalent)

4.5 Volumetric flat bottom boiling flask – 250 mL

4.6 Erlenmeyer flask – 500 mL

4.7 Volumetric pipettes – Class A

4.8 Vacuum filter flask with adapter – 2 Liter

4.9 Gooch crucibles – Coors #4

4.10 Glass fiber filter paper – 2.4 cm circles (Whatman 934-AH or equivalent)

4.11 Desiccator

4.12 pH meter

5. **Reagents and Supplies**:

5.1 Concentrated nitric acid (HNO_3)

5.2 Nitric Acid (1+1)

5.3 Aqueous ammonium hydroxide solution (1+1)

5.4 Ammonium Citrate – EDTA Solution

1. Dissolve 25g disodium EDTA (ethylenedinitrilotetraacetic acid) and 50g dibasic ammonium citrate in 1.5 L H_2O.
2. Nearly neutralize by adding 30 mL aqueous ammonium hydroxide (NH_4OH) solution.
3. Adjust the pH to 7.00 by adding (1+1) aqueous ammonium hydroxide solution.
4. Dilute to 2 L with H_2O.

5.4 Quimociac Reagent

1. Dissolve 70 g sodium molybdate dihydrate (Na_2MoO4. H_2O) in 150 mL H_2O.
2. Dissolve 60 g citric acid in a mixture of 85 mL concentrated HNO_3 and 150 mL H_2O. Allow to cool.
3. Gradually add the sodium molybdate solution to the citric acid - (HNO_3) mixture with stirring.
4. Dissolve 5 mL synthetic quinoline in a mixture of 35 mL HNO_3 and 100 ml H_2O.
5. Gradually add the quinoline solution to the molybdate-citric acid- (HNO_3) solution, mix, and let stand for 24 hours.
6. Filter.
7. Add 280 mL acetone, dilute to 1 L with H_2O, and mix.

6. **Instrument Calibration**:

Perform daily balance and oven temperature verification.

7. **Analysis**:

7.1. Thoroughly mix the sample before weighing by rotating and shaking the bottle. Weigh approximately 0.5 g sample into a 250 mL boiling flask. Record this weight to the nearest 0.0001 g.

7.2. Add ~100 mL ammonium citrate/EDTA solution to the flask and gently swirl to mix.

7.3. Secure the flask on the shaker rack in the 65°C ± 3°C water bath. Allow the flask to sit in the water bath for about 15 minutes to warm the solution to approximately 65°C.

7.4. Close the flask tightly with a rubber stopper and set the shaker to gently shake the flask for ~60 minutes.

7.5. Remove the flask from the water bath, fill the flask almost to the 250 mL mark with H_2O, and cool to room temperature.

7.6. Dilute the flask to volume with H_2O, tightly stopper, and mix. Allow the particulates to settle overnight, or the solution may be filtered to remove the particulates.

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

Guarantee	**Suggested Amount to pipet**
<9.7%	50 mL
9.7 – 19%	25 mL
>19%	15 mL

7.8. Add H_2O to bring the total volume to ~100 mL.

7.9. Add ~10 mL HNO_3 (1+1), place on a hot plate in a fume hood, and gently heat to boiling or near boiling for ~10 minutes. **Carefully swirl the flask several times during the heating process to prevent the solution from super heating.**

7.10 Remove from the hot plate and add ~50 mL Quimociac reagent.

7.11 Place back on the hot plate, and gently boil the precipitate solution for ~1 minute.

7.12 Remove from the hot plate and allow to cool to room temperature. Carefully swirl the flask 3-4 times during cooling.

7.13 Weigh a Gooch crucible which has been fitted with a glass fiber filter paper and previously dried at ~250°C. Record this weight to the nearest 0.0001 g.

7.14 Using the vacuum flask and vacuum, filter the precipitate into the Gooch crucible.

7.15 Wash the precipitate with five ~25 mL portions of H_2O, allowing each portion to drain thoroughly before adding the next portion.

7.16 Dry the crucible containing precipitate for ~30 minutes in a ~250°C oven.

7.17 Cool to room temperature in a desiccator.

7.18 Weigh the crucible and precipitate. Record this weight to the nearest 0.0001 g. If the weight of the precipitate is greater than 1.0 g, repeat steps 11.7 – 11.18 using a smaller aliquot.

8. **QA/QC**:

A Laboratory Control Sample (LCS) should be run with each set (12 samples or less). An acceptable LCS is a Magruder check sample with the reported mean and standard deviation for the direct available phosphorus as P_2O_5, (Magruder method code 041). An acceptable recovery for the LCS is to be within the Magruder mean ± 3 Magruder standard deviations. The minimum reportable result is 0.1%.

9. **Calculations**:

Calculate percent <u>available</u> phosphoric acid (P_2O_5):

$$\% P_2O_5 = \frac{\text{Weight of precipitate x DF x 0.03207 x 100}}{\text{Sample weight (g)}}$$

Where:

DF = dilution factor = 250mL/aliquot
0.03207 = Gravimetric factor derived from:
Molecular weight of P_2O_5 = 141.90
Molecular weight of QMP = $(C_9H_7N)_3H_3PO_4 \cdot 12MoO_3$ = 2212.71

$$\frac{P_2O_5}{2QMP} = \frac{141.90}{2 \times 2212.71} = 0.03207$$

10. **Reporting**:

10.1 The data packet will include the date of analysis, analyst, LCS, samples, measured weights, and the spreadsheet with the calculated results.

10.2 The Section Supervisor or designee will review the data packet and confirm the proper QC requirements were met. Either the Section Supervisor, designee, or analyst can enter the results on the laboratory sheets. If the analysis were performed by a seasonal, non-permanent employee, the Section Supervisor or designee shall enter the results on the laboratory sheet.

11. **References**:

Official Methods of Analysis of AOAC International, Methods 993.31 <u>(2.3.14) and 962.02 (2.3.03), 19th Edition, 2012</u>.

U.S.D.A. Food Safety and Inspection Service, Chemistry Laboratory Guidebook, Method 3.009, June 1987.

Approvals:

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Revision Log:

Date	What was Revised? Why?
8/23/19	Changed designation. Minor formatting. Updated reference. Fixed equation