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Minerals in Feed, Forage and Manure Samples
Dry Ashing Method

1. Application

In this procedure the minerals in feed, forage and manure samples are analyzed for phosphorus (P), potassium (K), magnesium (Mg), and calcium (Ca).

2. Summary of Methods

An Atomic Absorption Unit (AA) is used to determine levels of K, Ca and Mg, while a spectrophotometer is used for P.

3. Safety

All chemicals should be considered a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should be made available to all personnel involved in the chemical analysis.

4. Interferences

5. Sample Collection, Preservation, and Handling

All samples are dried at 55^{o}C in a cabinet-type forced air dryer for 12-18 hrs. After drying the sample is ground to pass through a 1 mm sieve.

6. Apparatus and Materials

6.1 Glass beakers (50ml)
6.2 Muffle furnace at 500^{o}C
6.3 Glass test tubes (20ml)
6.4 Erlenmeyer flasks (50ml)
6.5 Pipette bank (3ml and 10ml)
6.6 Pipette (1ml)
6.7 Atomic Absorption Unit (AA)
6.8 Spectrophotometer

7. Reagents

7.1 0.33 N Magnesium Acetate [Mg(OAc)2]
7.1.1 Dissolve 35.7 g of $Mg(C_2H_3O_2) \cdot 4H_2O$ in about 600 ml of distilled water.

7.1.2 Dilute to 1 liter.

7.2 Hydrochloric Acid (HCl)

7.2.1 Dilute 85 ml concentrated HCl to 1 liter distilled water

7.3 Vanadomolybdate (HNO_3)

7.3.1 Dissolve 2.50 g of NH_4VO_3 in 600 ml of hot distilled water. Cool and add 500 ml of concentrated HNO_3.

7.3.2 Dissolve 50 g of $(NH_4)_6MO_7O_{24} \cdot 4H_2O$ in 800 ml of distilled water.

7.3.3 Add to vanadomolybdate solution and dilute to 2 liters.

7.4 La Stock

7.4.1 Weigh out 23.456 g La_2O_3, slowly add 100 ml of concentrated HCl to dissolve (perform under hood, as there is fuming)

7.4.2 Add 0.1525 g NaCl and transfer quantitatively to 2 liter volumetric flask, diluting with distilled water up to volume.

8. Methods

Sample Preparation

8.1 Forages: Mix sample thoroughly, weigh one 0.50 g sub-sample from each sample into a small beaker.

8.2 Manures: Mix sample thoroughly, weigh one 0.25 g sub-sample from each sample into a small beaker. Note: when a 0.25 g sample is used readings on the AA and spectrophotometer are multiplied by two to adjust for the smaller initial sample weight. Other figures are recorded as is, unless a dilution is required.

8.3 Grains and High Fat Samples: Mix sample thoroughly and weigh two 0.50 g sub-samples from each sample into a small beaker. In one of the sub-samples (for phosphorus analysis) add 10 ml Magnesium Acetate. Put on hot plate, heat and evaporate to dryness. The other sub-sample (for Ca, Mg, and K analysis) is run as a regular forage sample, see below.

Step 1 for P, K, Mg, Ca

8.4 In muffle furnace, ash the forage and evaporated grain samples for 2 hours at 500°C.

8.5 After ashing, take out samples and let cool.

8.6 Add 10 ml HCl to small beakers, let stand 15 minutes.

Step 2 for P

8.7 Pipette a 1 ml aliquot from small beakers into small test tubes.

8.8 Add 3 ml Vanadomolybdate to each test tube and aliquot.

8.9 Add 10 ml distilled water to test tubes. Let color develop for at least 15-20 minutes, before reading on Spectrophotometer.

Step 2 for K, Mg, Ca

8.10 Add 20 ml distilled water to small beakers.

8.11 Pipette a 1 ml aliquot from beakers into small Erlenmeyer flasks.

8.12 Add 10 ml La Stock to Erlenmeyer flasks.

8.13 Add 20 ml distilled water to Erlenmeyer flasks.

Determination of mineral content

8.14 Small test tubes are used for reading P on the Spectrophotometer.

8.15 Erlenmeyer flasks are used for K, Ca, Mg readings on the Atomic Absorption Unit.

8.16 Remaining aliquot in small beakers may be used for sulfur analysis, see procedure "Sulfur Determination for Manures and Forage."

9. Calculations

9.1 Sample absorption is taken from spectrophotometer and AA.
9.2 Results are reported on a dry matter basis.
9.3 Manure phosphorus results are adjusted x2 when using a 0.25 g sample size.

10. Quality Control

10.1 Laboratory Reagent Blank (LRB) – At least one LRB is analyzed with each batch of samples to assess contamination from the laboratory environment. Contamination from the laboratory or reagents is suspected if LRB values exceed the detection limit of the method. Corrective action must be taken before proceeding.
10.2 Standard – One or more standards of known mineral content are analyzed with each batch of samples to check instrument calibration and procedural accuracy.

11. Reporting

Results are reported as a percent of dry matter basis.

12. References

12.1 Wisconsin Procedures for Soil Testing, Plant Analysis and Feed & Forage Analysis, No. 6, Soil Fertility Series; last revised 1987 by E.E. Schulte, J.B. Peters and P.R. Hodgson.