

## **Sulfur Determination in Manure and Forage**

### **1. Application**

This procedure measures the concentration of sulfur in feed, forage and manure samples.

### **2. Summary of Methods**

A turbidimeter is used to determine levels of sulfur in these materials.

### **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**

The turbidimetric procedure is sensitive to variations in temperature and humidity.

### **5. Sample Collection, Preservation, and Handling**

All samples are dried at 55°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 See procedure "Minerals in Feed, Forage and Manure Samples."
- 6.2 Funnel tubes (25ml)
- 6.3 Colorimeter tubes (20ml)
- 6.4 Turbidimeter

### **7. Reagents**

- 7.1 See procedure "Minerals in Feed, Forage and Manure Samples" for partial list of reagents.
- 7.2 Sulfur Extract [ $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ ]
  - 7.2.1 Dissolve 38.6 g of Calcium Phosphate [ $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ ] in approximately 15 liters of distilled water.
  - 7.2.2 Add 2185 ml of Glacial Acetic Acid and dilute to 19 liters.
- 7.3 Sulfur Developer [ $\text{BaCl}_2\text{-HOAC}$ ]

- 7.3.1 Dissolve 47.5 g of Gum Arabic in approximately 4.5 liters of hot distilled water.
- 7.3.2 Filter through suction funnel with filter paper #1, twice.
- 7.3.3 Once clear add 475 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ .
- 7.3.4 Add 4275 ml of Glacial Acetic Acid and dilute to 9.5 liters.

## **8. Methods**

- 8.1 See procedure "Minerals in Feed, Forage and Manure Samples." This procedure picks up after "Step 2 for K, Mg, Ca," using the aliquot remaining in the small beakers.
- 8.2 Pipette a 2 ml aliquot from small beakers into 25 ml funnel tubes. Add 18 ml sulfur extract solution.
- 8.3 Pipette 10 ml from funnel tubes into the colorimeter tubes.
- 8.4 Add 10 ml of sulfur developer solution to colorimeter tubes using an automatic pipetter.
- 8.5 Let side for 10-15 minutes (until the bubbles have settled.)
- 8.6 Read using turbidimeter. Set the blank in the meter and check the standard, adjusting to correct reading if needed.
- 8.7 Continue with reading the samples.

## **9. Calculations**

- 9.1 Forage % Sulfur = reading on turbidimeter \* 2 \* 0.01288
- 9.2 Manure % Sulfur = reading on turbidimeter \* 2 \* 0.01288 \* 2

## **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.