

	FM-806	
	FERTILIZER METHODS	Chapter
		SECONDARY/MICRONUTRIENT ANALYSIS
		Subject
	Sulfur – Free	

SCOPE: This is an analytical procedure for the determination of free sulfur (elemental sulfur) in mixed fertilizer by gravimetric method.

PRINCIPLE: Free sulfur is determined by a soxhlet extraction with carbon disulfide (CS₂.) The percent sulfur is determined after drying by weight difference.

SAFETY: Each laboratory is responsible for maintaining a current file of the Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

APPARATUS & EQUIPMENT:

- Cellulose extraction thimbles (25 mm X 80 mm) Whatman Cat. # 2800258 or equivalent
- 125 mL boiling flask with appropriate glass joint
- 30 mm extraction tube with appropriate glass joint
- 30 mm extraction condenser with appropriate glass joint
- Elbow joint with appropriate glass joint
- Water bath w/thermolyne hot plate or equivalent
- Analytical balance (.0001 g)
- Drying oven (100°C)
- Fume hood

REAGENTS & CHEMICALS:

- Deionized (D.I.) water
- Carbon disulfide (CS₂) (Reagent Grade A.C.S.)
- Carbon tetrachloride (CCl₄) (99% Anhydrous)
- Carbon disulfide Extraction Solution:
60% CS₂ + 40% CCl₄. To make a 1 L solution, combine 600 mL of CS₂ and 400 mL of CCl₄.

NOTE: Sulfur-coated urea tends to give elevated results in free sulfur analysis. It leaves a brownish, oily residue in the boiling flask after distillation.

**SAMPLE
PREPARATION:**

NOTE ON WEIGHING SAMPLE:

% Guaranteed	Approximate Weight + or - .1 g
< 10 %	2.0 g
>= 10 %	.5 g

1. Weigh appropriate sample directly into cellulose thimble and record weight (SW).
2. Weigh and record initial weight of clean, dry 125 mL boiling flask (TW).
3. Rinse sample in cellulose thimble with at least 3 (20 mL aliquots) of 65° C D.I. water.
4. Dry thimbles in oven (100°C) for at least 1 hour, or until thimble is dry. Moisture left in thimble will interfere with extraction process.
5. Add approximately 65 mL of CS₂ extraction solution to boiling flask, place thimble in the extraction tube (column) and add approximately 15 mL of the CS₂ extraction solution to the thimble in the extraction column.
6. Connect extraction tube (reflux column) to boiling flask.
7. Place reflux column w/boiling flask on hot plate and reflux for 2-3 hours.
8. After refluxing, remove reflux column w/boiling flask and drip dry the thimble in reflux column. Remove thimble from reflux column and place in corresponding beaker.
9. Disconnect column from boiling flask and pour remaining CS₂ extraction solution into the boiling flask.
10. Place boiling flask on distilling apparatus using elbow joint.
11. Distill and collect CS₂ solution for reuse.
12. Remove the boiling flask from distilling apparatus and air dry. Place flask in drying oven for 15-30 minutes. A yellowish residue in flask indicates the presence of elemental sulfur.
13. Weigh flask (FW) and calculate.

CALCULATIONS:

S = Free Sulfur

FW = Final weight of flask

TW = Tare weight of flask

SW = Sample weight

%S(Free Sulfur) = $((FW) - (TW)) / (SW) * 100$

APPROVAL:

Approved by: Leigh Humphreys Date: 1/2/03
Signature

Bureau Chief
Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	6/12/98	Replaces S121	J, Corry
Revised	01/02/03		G. H. Huang

REFERENCE:

AOAC 15th Edition, *Method 980.02*