

FERTILIZER METHODS

Chapter

BORON ANALYSIS

Subject

Boron-Continuous Segmented Flow

SCOPE: This is an automated analytical procedure for the determination of boron in fertilizer samples. Sample range is based on a 1 g sample diluted to 250 mL. Different ranges are possible by varying the sample weight and/or dilution volume.

PRINCIPLE: The percent of boron is determined on the basis of a colorimetric reaction of the azomethine-H dye, ammonium acetate buffer and the boron present. **This procedure is not applicable in the presence of greater than 5% urea or urea-formaldehyde resins. The urea-formaldehyde tends to bleach the color complex, yielding an erroneously low percent found.**

SAFETY: Each laboratory is responsible for maintaining a current file of the Occupational Health and Safety Act (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:

- Balance (accuracy to 0.001 g)
- Acid fume hood
- Hot plate, Thrifty or equivalent
- Wesco Seraclear filter or equivalent
- Culture tube, 16 x 100 mm or equivalent to fit Wesco Seraclear filter
- Flasks, 250 mL, 500 mL and 1 L volumetric (class "A")
- Pipets, 15 mL, 25 mL, and 50 mL volumetric (class "A")
- Automated segmented continuous flow analyzer with air injector
- Automatic sampler – with adjustable sample and wash periods
- Proportioning Pump
- Analytical manifold with heater
- Colorimeter equipped with 420 nm filter and 30 mm x 1.5 mm id. flow cell

- Chart recorder, compatible with colorimeter output
- Pump tubes (See Fig. 1 for sizes)
- Tygon tubing (0.030 I.D.)
- Sample cups, to fit Automatic sampler

REAGENTS & CHEMICALS:

- Deionized water (D.I.)
- Sulfuric acid, (Certified A.C.S. grade) **Caution – strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment (H₂SO₄)**
- Phosphoric acid, (85% H₃PO₄ HPLC grade) **Caution – strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment**
- Activated carbon – decolorizing (Darco G60 or equivalent)
- Ammonium acetate (Certified A.C.S. grade)
- Ascorbic acid (Certified A.C.S. grade)
- Azomethine-H Dye
- Barium chloride (Certified A.C.S. grade)
- Boron Certified Reference Solution (1000 ppm)
- Brij-35 stock solution (30%)
- Disodium EDTA (Certified A.C.S. grade)
- Brij-35 wetting solution (15%)

Brij-35 stock solution (30%) 250 mL

Deionized water 250 mL

Add 250 mL of 30% Brij-35 stock solution to 250 mL of deionized water. Mix well.

- Ammonium acetate buffer solution

Ammonium acetate 500 g

Disodium EDTA 10 g

Deionized water 1800 mL

Brij-35 (15%) 25-30 drops

Dissolve 500 g of ammonium acetate (98%) and 10 g of disodium EDTA (99%) in 1800 mL of deionized water. Adjust pH to 5.25 by adding small amounts of concentrated sulfuric acid. Add 25-30 drops of Brij-35 (15%) wetting agent and bring to final volume (2 liters) with deionized water and mix well.

- Barium chloride solution (BaCl₂)

Barium chloride	100 g
Deionized water	800 mL

Weigh 100 g of barium chloride in 800 mL of deionized water. Bring to final volume (1 L) with deionized water and mix well.

- Azomethine-H boron reagent

Ascorbic acid	5.0 g
Azomethine-H Dye	1.0 g
Deionized water	100 mL
Brij-35 (15%)	3-5 drops

Weigh 5.0 g of ascorbic acid into a 250 mL volumetric flask and add 100 mL of deionized water. Add 1.0 g of azomethine-H to the solution and add 3-5 drops of 15% Brij-35 wetting agent. Bring to volume with deionized water and mix well.

NOTE: The coloring reagent is light sensitive and should be made up weekly and kept refrigerated when not in use.

- 50 ppm Boron stock standard

Boron Certified Reference Solution	25 mL
Deionized water	475 mL

Pipet 25 mL of boron Certified Reference Solution into a 500 mL volumetric flask containing 400 mL of deionized water. Bring to volume with the deionized water and mix well

- Boron working standard (high-range, 10 ppm)

50 ppm boron Certified Reference Solution	50 mL
Deionized water	200 mL

Pipet 50 mL of 50 ppm boron Certified Reference Solution into a 250 mL volumetric flask containing 200 mL of deionized water. Mix well.

- Boron working standard (mid-range, 5 ppm)

50 ppm boron Certified Reference Solution	25 mL
Deionized water	225 mL

Pipet 25 mL of 50 ppm boron Certified Reference Solution into a 250 mL

volumetric flask containing 225 mL of deionized water. Mix well.

- Boron working standard (low-range, 3.0 ppm)

50 ppm boron Certified Reference Solution 15 mL

Deionized water 235 mL

Pipet 15 mL of 50 ppm boron Certified Reference Solution into a 250 mL volumetric flask containing 235 mL of deionized water. Mix well.

- System rinse / wash solution

Deionized water 800 mL

15% Brij-35 wetting agent 4 drops

Add 4 drops of 15% Brij-35 wetting agent to 800 mL of deionized water and mix well

SAMPLE HANDLING:

For samples containing a considerable amount of “free-floating” trash in the flasks, filter a portion of the sample in a culture tube using a Wesco Seraclear filter. For samples that are highly colored, add approximately 1-10 g of activated carbon to the flask after the sample has been brought to volume and shake well, use a Wesco Seraclear filter to remove any residue before analyzing.

NOTE: For all deficient samples, add a few drops of 85% phosphoric acid and 3-5 mL of 10% barium chloride to samples before placing them on the hot plate. The phosphoric acid breaks down the urea-formaldehyde complex. The barium chloride addition step precipitates any sulfates or phosphates which may interfere with the colorimetric analysis of boron.

SAMPLE PREPARATION:

Weigh the appropriate weight directly into 250 mL volumetric flask (See Table 1).

Table 1

%B	Approximate Weight +/- .10 g	Dilution
< 0.03%	3.00 g	None
0.03 – 0.08%	2.50 g	None
0.08 – 0.16%	1.25 g	None
0.16 – 0.25%	0.80 g	None
0.25 – 0.40%	0.50 g	None
>0.40%	0.50 g	Make appropriate dilution as needed

1. Add 100 mL of deionized water to each sample.
2. Place the samples on hot plate and digest at slow boil for 30 minutes.
3. Allow the samples to cool to room temperature and bring to volume with deionized water.
4. Shake well. **NOTE: 30-45 seconds of vigorous shaking may be required to mix sample completely.**

**SAMPLE
ANALYSIS:**

See “FM-801 Sample Analysis S.O.P.” for Instrument Conditions

CALCULATIONS:

Drift Corrected Readings:

Where:

R = sample reading between standards S_{m1} ... and S_{m2}

S_{m1} = reading of medium standard preceding the sample

S_{m2} = reading of second medium standard following the sample

T(R) = set position number of the sample

T(S_{m1}) = set position number of the medium standard preceding the sample

T(S_{m2}) = set position number of the second medium standard following the sample

S.W. = sample weight

$$\text{Corrected Reading} = [R - (S_{m1}) - (50)] - \frac{(S_{m2}) - (S_{m1})}{T(S_{m2}) - T(S_{m1})} \times [T(R) - T(S_{m1})]$$

$$\% \text{Boron} = \frac{[(\text{corrected chart reading})(5 \text{ ppm})(250 \text{ mL})]}{[(S.W.)(50)(10^6 \text{ mg/g})]} (100)$$

(50 – represents 50% full scale from the 5 ppm medium boron standard)

QA/QC:

A boron internal standard and a deionized water blank are analyzed with each set to insure the accuracy and precision of the method.

APPROVAL:

Approved by: Leigh Humphreys Date: 4/10/02
 Signature

Bureau Chief
 Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	06/12/98	Replaces M-100	J. Corry
Revised	04/10/02		G. H. Huang

REFERENCE:

AOAC 16th Edition, *Method 982.01*

BORON SYSTEM

Fig. 1

