Nitrogen, ammonia plus organic, titrimetric, digestion-distillation

Parameter and Code:
Nitrogen, ammonia plus organic, total-in-bottom-material, dry wt, I-5553-85 (mg/kg as N): 00626

1. Application

This method may be used for analysis of bottom material containing at least 10 mg/kg of total ammonia plus organic nitrogen. Only that portion of bottom material that passes a 2-mm sieve is taken for analysis (method P-0810, sub-sampling, bottom material, coring).

2. Summary of method

The sample is subjected to a digestion whereby all organic nitrogen-containing compounds are converted to ammonium salts. The resulting mixture is then made strongly alkaline, and the ammonia so formed is distilled from the mixture into a solution of boric acid and subsequently determined by titration with standard sulfuric acid solution.

3. Interferences

There are no known interferences with this method.

4. Apparatus

Kjeldahl distillation apparatus, 500-mL flasks.

5. Reagents

5.1 *Ammonium chloride,* crystals.

5.2 *Boric acid solution,* 20 g/L: Dissolve 20 g H_3BO_3 crystals in 800 mL ammonia-free water and dilute to 1 L.

5.3 *Digestion catalyst:* Tablets containing 3.5 g K_2SO_4 and 0.175 g HgO (Scientific Chemical Technical Sales Inc., SCT Kel-catalyst No. KC-M3, or equivalent.

5.4 *Mixed indicator solution:* Dissolve 20 mg methyl red and 100 mg bromocresol green in 100 mL 95-percent ethanol. Store in a well-sealed bottle.

5.5 *Sodium carbonate solution,* 0.0357*N*: Dissolve 1.892 g primary standard Na_2CO_3 in carbon dioxide-free water and dilute to 1,000 mL.

5.6 *Sodium hydroxide-thiosulfate solution: Cautiously,* dissolve 500 g NaOH in 600 mL ammonia-free water. Add 80 g $Na_2S_2O_3 \cdot 5H_2O$ and dilute to 1 L.

5.7 *Sodium thiosulfate,* crystals, $Na_2S_2O_3 \cdot 5H_2O$.

5.8 *Sucrose.*

5.9 *Sulfuric acid,* concentrated, sp gr 1.84.

5.10 *Sulfuric acid standard solution,* approx. 0.036*N*: *Cautiously,* add 1.0 mL concentrated H_2SO_4 (sp gr 1.84) to 800 mL ammonia-free water and dilute to 1 L. Standardize by titrating 25.0 mL 0.0357*N* Na_2CO_3 to pH 4.5. Compute normality of sulfuric acid standard solution to four decimal places.

6. Procedure

6.1 Free the distillation apparatus of ammonia by boiling ammonia-free water until the distillate shows no trace using nessler reagent----- **CAUTION**: **deadly poison**.

6.2 Weigh to the nearest milligram, 3 g of bottom-material sample, prepared as directed in method P-0810, and transfer to the digestion flask.

6.3 In the same manner prepare a blank, using 2.0 g sucrose. Analyze the blank and each sample as directed in paragraphs 6.4 to 6.10.

6.4 *Cautiously,* add 25 mL concentrated H_2SO_4 (sp gr 1.84), and under a hood, swirl the contents of the flask until thoroughly mixed.

6.5 Add three digestion-catalyst tablets and mix well. Add a few glass beads and begin the digestion. Continue the digestion until a clear solution is obtained, and then continue fuming 1 h.

6.6 Cool the flask until crystals appear (do not cool completely). Add 150 mL ammonia-free water; mix and allow to cool.

6.7 Add 100 mL NaOH-$Na_2S_2O_3$ solution. Immediately connect the flask to the distillation apparatus and *cautiously* mix the contents by swirling.

6.8 Distill at a rate of not more than 10 mL/min and no less than 6 mL/min; collect the distillate in a 250-mL volumetric flask containing 25 mL boric acid solution. The tip of the delivery tube must be below the surface of the boric acid solution in the receiving flask.

6.9 Collect approx. 200 mL of distillate, dilute to 250 mL with ammonia-free water, and mix.

6.10 To the distillate, add 3 drops mixed indicator solution, and titrate with sulfuric acid standard solution until the color of the solution changes from yellow to red.

7. Calculations

Nitrogen, ammonia plus organic (mg/kg) =

$$\frac{V_a \times N_a \times 14{,}000}{Wt_s}$$

where

V_a = volume of standard H_2SO_4 used to titrate sample, milliliters, minus volume used to titrate blank, milliliters,

N_a = normality of standard H_2SO_4 solution, and

Wt_s = weight of sample, grams.

8. Report

Report nitrogen, ammonia plus organic, total-in-bottom-material (00626), concentrations as follows: 10 to 100 mg/kg, nearest 1 mg/kg; 100 mg/kg and above, two significant figures.

9. Precision

Precision data are not available for this method.