AMMONIA NITROGEN

PRINCIPLE

The sample is buffered at a mildly alkaline pH by an excess of magnesium oxide, liberating ammonia from a portion of any ammonium salts present in solution. Continuous distillation removes the ammonia, shifting the equilibrium and allowing quantitative recovery of the ammonia by absorption in an excess of standard acid solution, without significant decomposition of other nitrogen-containing materials.

SCOPE

The method is applicable to light and heavy steepwater (Note 1) and most organic and inorganic materials. Nitrogenous materials which are volatile in steam will interfere.

SPECIAL APPARATUS

Standard Kjeldahl distillation equipment with 800-mL Kjeldahl flasks and suitable connecting bulbs are recommended.

REAGENTS

1. Magnesium Oxide: Reagent grade, carbonate-free

2. Sulfuric Acid Solution, 0.1 *N*: Standard (Note 2)

3. Sodium Hydroxide Solution, 0.1 *N*: Standard (Note 2)

4. Methyl Red-Bromcresol Green Indicator: Dissolve 0.33 g bromcresol green and 0.66 g methyl red dyes in 1 L of 95% ethyl alcohol. Add sufficient 0.1 *N* sodium hydroxide solution to produce a green color; add dropwise just sufficient 0.1 *N* hydrochloric acid to produce a deep wine-red color.

5. Anti-Bump Material: Boiling chips, glass beads or other suitable material

6. Anti-Foam Agent

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SAFETY

Person(s) performing this method should wear appropriate protective equipment. Glassware should be carefully inspected for defects before use. Analysts should also review the Manufacturer's Safety Data Sheets (MSDS) for all of the chemicals required; however this review is not a substitute for adequate laboratory safety training.

PROCEDURE

Measure accurately an excess of standard 0.1 *N* sulfuric acid solution (25 mL usually sufficient) into a 500-mL Erlenmeyer flask. Add about 25 mL of purified water and connect flask to distillation assembly so that condenser delivery tube is immersed in absorbing acid (Note 3).

Weigh 20.0 g (± 0.1 g) of heavy steepwater (Note 4) into a 250-mL beaker. Dilute to about 100 mL volume; add 1 *N* sodium hydroxide solution to adjust to pH 7.0 (± 0.5), as measured with a pH meter or a suitable indicator.

Transfer the neutralized sample (Note 5) quantitatively to an 800-mL Kjeldahl flask with the help of purified water, and dilute to a total volume of about 350 mL. Add 2 or 3 boiling chips or glass beads, 3-5 g of antifoam agent and 2 g of magnesium oxide. Connect flask to condenser by means of connecting bulb, turn on heater, and mix contents of flask by swirling. Distill at a moderate rate (Note 6) until all ammonia has passed into the absorbing solution (100 mL of distillate collected normally).

Remove receiving flask from distillation assembly, add about 0.25 mL of methyl red-bromcresol green mixed indicator (Note 7), and titrate with 0.1 *N* standard sodium hydroxide solution to a permanent green end point.

Make a blank determination on all reagents, substituting purified water for the sample, and calculate the 0.1 *N* sulfuric acid equivalence (blank).

CALCULATION

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$$\text{\% Ammonia Nitrogen (as is)} = \frac{(\text{mL } 0.1\ N\ H_2SO_4 - \text{Blank} - \text{mL } 0.1\ N\ \text{NaOH}) \times}{\text{Sample Wt. (g)}}$$

NOTES AND PRECAUTIONS

1. The steeping (soaking) of corn in a dilute sulfurous acid solution extracts solubles including carbohydrates, and the latter are largely fermented to lactic acid during the process. The concentrated extract is known as heavy steepwater in the corn wet milling industry, and it is designated Condensed Fermented Corn Extractives by the American Association of Feed Control Officials. The latter organization provides the following definition:

   "Condensed Fermented Corn Extractives is the product obtained by the partial removal of water from the liquid resulting from steeping corn in a water and sulfur dioxide solution which is allowed to ferment by the action of naturally occurring lactic acid producing microorganisms as practiced in the wet milling of corn."

2. Standard solutions of sulfuric acid and sodium hydroxide at concentrations of 0.04 *N* can be used to increase sensitivity of the method, when the ammonia nitrogen concentrations are unusually low.

3. Fifty milliliters of 0.4% aqueous boric acid solution may be used alternatively for absorption of ammonia. In this case, however, the titration is dilution-sensitive and distillate volumes (samples and blanks) should be adjusted to constant values by addition of purified water prior to titration. The boric acid solution containing ammonia is titrated directly with standard sulfuric acid solution, eliminating the use of standard alkali as in the normal procedure. If ammonia is absorbed in boric acid solution, increase the quantity of indicator for a sharper end point.

4. The 20-g sample of steepwater usually contains about 20 mg of ammonia nitrogen. If the sample contains substantially more or less ammonia nitrogen, adjust sample weight accordingly.

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5. The sample solution must be essentially neutral when the magnesium oxide is added. An excess of acid might consume all of the oxide, while an excess of alkali might cause liberation of ammonia from organic compounds.

6. Steepwater samples tend to foam during distillation and care must be exercised to avoid carry over of sample solution into the receiver which invalidates the test.

7. Methyl red or methyl red-methylene blue mixed indicators may also be used.

METHOD HISTORY

Steepwater, Ammonia Nitrogen (J-12), Date of Acceptance 5-15-1962, Revised 3-05-2004.