

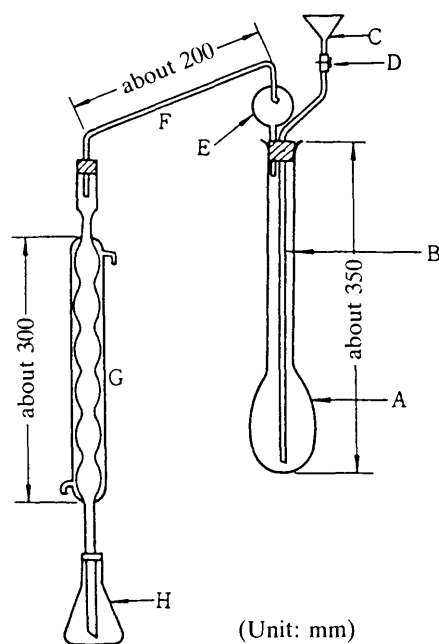
B. GENERAL TESTS

Nitrogen Determination

The Nitrogen Determination Test is designed to quantify ammonia in ammonium sulfate obtained by decomposing organic substances containing nitrogen with sulfuric acid.

(1) Kjeldahl Method

Apparatus Use the apparatus illustrated in the figure below. Ground-glass surfaces may be used for joints.



- A: Kjeldahl flask (Made of hard glass. Capacity: about 300 ml)
- B: Glass tube
- C: Funnel for addition of alkaline solution
- D: Rubber tube (Coupling of B and C. With pinch cock)
- E: Spray trap
- F: Delivery tube
- G: Condenser
- H: Absorption flask (Capacity: about 300 ml)

Procedure Unless otherwise specified, proceed as directed below.

Weigh accurately a quantity of the sample corresponding to about 20 to 30 mg of nitrogen, place it into Kjeldahl flask A, and add 5 g of powdered potassium sulfate, 0.5 g of cupric sulfate, and 20 ml of sulfuric acid. Tilt A at about 45°, heat gently until effervescence almost stops, and raise the temperature to boil. After the contents become a clear, blue solution, heat for another 1 to 2 hours. Cool, add gradually 150

B. GENERAL TESTS

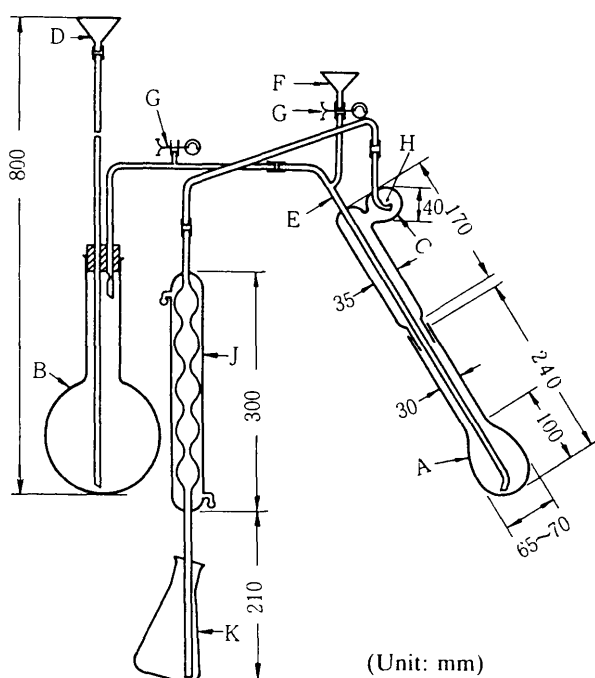
ml of water, and cool again. Add 2 or 3 granules of boiling chips or granulated zinc, and assemble the apparatus.

Measure exactly 25 ml of 0.05 mol/l sulfuric acid, transfer into absorption flask H, add about 50 ml of water, and immerse the lower end of condenser G into this solution. Add gradually 85 ml of sodium hydroxide solution (2 5) through funnel C, wash with a small quantity of water, close the pinch cock on rubber tube D, mix the contents by lightly shaking A, and heat gently. When it starts to boil, turn up the heat, and distill until about two thirds of the contents are distilled. Remove the lower end of G from the solution in H, continue the distillation for a short time, wash the lower end of G with a small quantity of water, and titrate the excess acid in the solution in H with 0.1 mol/l sodium hydroxide solution. The endpoint is usually confirmed using a potentiometer. When an indicator (3 drops of bromocresol green - methyl red mixture TS) is used, the endpoint is the time when the color of solution changes from red-purple through pale grayish yellow to pale grayish green. Perform a blank test and make any necessary correction.

1 ml of 0.05 mol/l sulfuric acid = 1.4007 mg of N

(2) Semi-micro Kjeldahl Method

Apparatus Use the apparatus made of hard glass, illustrated below. Ground glass may be used for joints. All rubber parts used in the apparatus should be boiled in sodium hydroxide solution (1 25) for 10 to 30 minutes and then in water for 30 to 60 minutes, and finally washed thoroughly with water before use.



A: Kjeldahl flask

B: Steam generator (Fill with water containing 2 to 3 drops of sulfuric acid.

B. GENERAL TESTS

Place boiling chips to prevent bumping.)

- C: Spray trap
- D: Water supply funnel
- E: Steam tube
- F: Funnel for addition of alkaline solution
- G: Rubber tube with a pinch cock
- H: Small hole (The diameter is approximately equal to the internal diameter of the tube.)
- J: Condenser (The lower end is beveled.)
- K: Absorption flask

Procedure Unless otherwise specified, proceed as directed below.

Weigh accurately or pipet a quantity of the sample corresponding to 2 to 3 mg of nitrogen, and place in Kjeldahl flask A. Add 1 g of a powdered mixture of 10 g of potassium sulfate and 1 g of cupric sulfate. Wash down the sample adhering to the neck of A with a small quantity of water. Add 7 ml of sulfuric acid, allowing it to flow along the inside wall of A.

Then, while shaking A, add cautiously 1 ml of hydrogen peroxide drop by drop along the inside wall of A. Heat A on a ceramic gauze or ceramic plate, over a free flame until the solution exhibits a clear blue color and the inside walls of A are free from carbonaceous material, and heat for another 1 to 2 hours. If necessary, cool, add a small quantity of hydrogen peroxide, and heat again. Cool, add cautiously 20 ml of water, cool the solution, and connect A to the distillation apparatus, washed previously by passing steam through it. To absorption flask K, add 15 ml of boric acid solution (1 : 25), and sufficient water to immerse the lower end of condenser tube J. Add 30 ml of sodium hydroxide solution (2 : 5) through funnel F, wash cautiously the funnel with 10 ml of water, immediately close the pinch cock attached to rubber tube G, and distill with steam until the distillate measures 80 to 100 ml. Remove the lower end of J from the solution, continue the distillation for a short time, wash the lower end of J with a small quantity of water, and titrate the distillate with 0.005 mol/l sulfuric acid. The endpoint is usually confirmed using a potentiometer. When an indicator (3 drops of bromocresol green-methyl red mixture TS) is used the endpoint is the time is when the color of the solution changes from red-purple through pale grayish yellow to pale grayish green. Perform a blank test in the same manner, and make any necessary correction.

1 ml of 0.005 mole/l sulfuric acid = 0.14007 mg of N