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APPENDIX B

(Clause 3.2, and Table 1)

METHODS OF TEST FOR AMMONIA, LIQUOR

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE 1 — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

NOTE 2 — Lunge-Ray pipette is recommended for carrying out analysis of the material.

B-2. DETERMINATION OF AMMONIA

B-2.0 Ammonia may be determined by either of the two methods, namely, Method A and Method B. In case of dispute Method B shall be adopted.

B-2.1 Method A

B-2.1.1 Reagents

B-2.1.1.1 Standard sulphuric acid — 0.5 N.

B-2.1.1.2 Methyl red indicator — Dissolve 0.5 g of water soluble methyl red in water and dilute the solution to one litre.

B-2.1.1.3 Caustic soda solution — 0.5 N, freshly standardized.

B-2.1.2 Procedure — Accurately weigh a 15 ml capacity ground glass stoppered weighing bottle containing 10 to 12 ml of water. Open the ammonia bottle and insert a clean and dry glass tube of about 5 mm bore to half the height of the liquid and transfer 1 to 1.5 ml of the material to the weighing bottle immediately replace the glass stopper and weigh. Place the weighing bottle in about 200 ml of water containing 50 ml of 0.5 N sulphuric acid and a few drops of methyl red indicator.

Due to the mass of the water and ammonia contained in the bottle, it will remain immersed in the acid. Open the stopper under the acid by manipulating with a glass rod and titrate the excess of acid with standardized caustic soda solution to a faint yellow end point.

B-2.1.2.1 Carry out a blank with all the reagents used for the test.

*Specification for water for general laboratory use (second revision).



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B-2.2.3 Procedure — Weigh the glass ampoule to the nearest 0.01 g. Gently heat the spherical part of the ampoule over a flame and dip the capillary end of the ampoule into the bottle containing the laboratory sample. Ensure that the ampoule is almost completely filled during cooling.

Withdraw the ampoule and dry the capillary tube carefully with filter paper. Seal the end of the capillary tube, without loss of glass, with an oxidizing flame. Allow the capillary tube to cool, wash it with water and wipe it carefully with filter paper.

Weigh the sealed ampoule accurately and calculate, by difference, the mass of the test portion.

B-2.2.3.1 Carefully place the ampoule containing the test portion into a 500 ml conical flask, fitted with a ground glass stopper, to which 50 ml of the boric acid solution, about 250 ml of water and several drops of the methyl red solution have already been added.

Stopper the conical flask and shake carefully so as to break the ampoule.

Unstopper the flask, rinse the stopper with water collecting the washings in the same flask.

Using a glass rod, grind the pieces of the ampoule, in particular those parts of the capillary tube which may have remained unbroken. Remove the glass rod, rinse it with water, collecting the washings in the same flask.

Titrate with the sulphuric acid solution until the indicator changes from yellow to red.

B-2.2.4 Calculation

$$\text{Ammonia, percent by mass} = \frac{1.703 V}{M}$$

where

V = volume in ml of standard sulphuric acid used, and

M = mass in g of the material taken for the test.

B-3. DETERMINATION OF RESIDUE ON EVAPORATION

B-3.1 Procedure — Measure 500 ml of the material in case of AR grade and 100 ml for technical grade in a volumetric flask. Add the whole material, in small portions, to a tared platinum crucible or other suitable dish, and evaporate to dryness on a steam bath, inside a fume cupboard. Dry the residue at $105 \pm 2^\circ\text{C}$ to constant mass.



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B-2.1.3 Calculation

$$\text{Ammonia, percent by mass} = \frac{1.703 (V_1 - V_2) N}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used in the blank,

V_2 = volume in ml of standard sodium hydroxide solution used in the test with the material,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

B-2.2 Method B

B-2.2.1 Reagents

B-2.2.1.1 Boric acid — 2 percent.

B-2.2.1.2 Standard sulphuric acid solution — 0.5 N.

B-2.2.1.3 — Methyl red — 0.1 percent. Dissolve 0.1 g of methyl red in 95 percent (v/v) ethanol and make up to 100 ml with the same ethanol.

B-2.2.2 Apparatus

B-2.2.2.1 Spherical glass ampoule — of thin glass, of suitable capacity and shape, for example, about 20 mm diameter, with one capillary end about 50 mm in length (a typical example is shown in Fig. 1).

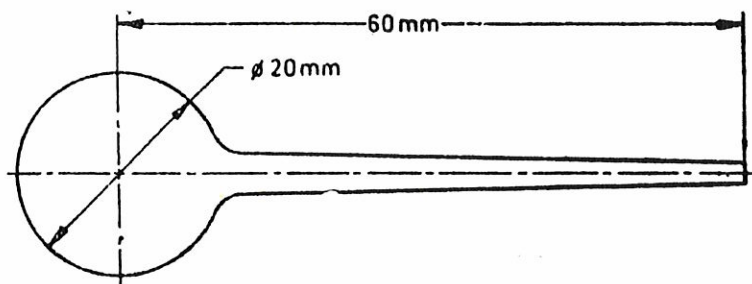


FIG. 1 SPHERICAL GLASS AMPOULE



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B-3.2 Calculation

$$\text{Residue on evaporation, percent by mass} = \frac{100 M}{VS}$$

where

M = mass in g of the residue,

V = volume in ml of the material taken for the test, and

S = relative density of the material.

B-4. DETERMINATION OF CARBONATE

B-4.1 Apparatus

B-4.1.1 *Nessler Cylinders* — 25 ml capacity.

B-4.2 Reagents

B-4.2.1 *Barium Hydroxide Solution* — saturated.

B-4.2.2 *Standard Sodium Carbonate (Anhydrous) Solution* — 0.01 N.

B-4.3 Procedure — Weigh accurately 1.5 g of the material, dilute to 20 ml with carbon dioxide-free water and add 5 ml barium hydroxide solution. Carry out a control test in the other Nessler cylinder by taking 0.5 ml of standard sodium carbonate solution, 20 ml of carbon dioxide-free water and 5 ml of barium hydroxide solution.

B-4.3.1 The material shall be taken to have passed the test if the turbidity produced in the test with the material is not greater than that produced in the control test.

B-5. DETERMINATION OF CHLORIDES

B-5.1 Apparatus

B-5.1.1 *Nessler Cylinders* — 50 ml capacity.

B-5.2 Reagents

B-5.2.1 *Silver Nitrate Solution* — 0.1 N approximately.

B-5.2.2 *Dilute Nitric Acid* — 4 N approximately.

B-5.2.3 Standard Chloride Solution A — Dissolve 1.648 g of sodium chloride (dried at $105 \pm 2^\circ\text{C}$) in water and dilute to 1 000 ml in a volumetric flask. One millilitre of this diluted solution contains 1.0 mg of chloride (as Cl).



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PRODUCT SPECIFICATIONS

PRODUCT	:	<i>LIQUOR AMMONIA (Ammonia Solution)</i>
DESCRIPTION	:	<i>A Clear Colorless Liquid Odor : Strongly Pungent & Characteristic</i>
SOLUBILITY	:	<i>Mixable with Water in all Proportions</i>
IDENTIFICATION	:	<i>I.P. Identification test for Ammonia – Positive</i>
RESIDUE ON EVAPORATION	:	<i>0.0068%</i>
SPECIFIC GRAVITY @ 25 C	:	<i>0.905 gms</i>
CHLORIDE (AS CL)	:	<i>Less than 0.0005%</i>
SULPHATE (AS SO ₄)	:	<i>Less than 0.0002%</i>
IRON (AS Fe)	:	<i>Less than 0.00002%</i>
CARBONATE (as CO ₃)	:	<i>Less than 0.002%</i>
ASSAY	:	

Liquor Ammonia confirms to IS: 799 - 1985

For Hyderabad Ammonia & Chemicals Pvt. Ltd.

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