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BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

Draft Indian Standard

Ammonium nitrate for explosives — Specification

(*Second Revision*)

भारतीय मानक मसौदा

विस्फोटकों के लिए अमोनियम नाइट्रेट — विशिष्टि

(*दूसरा पुनरीक्षण*)

ICS 71.100.30

Explosives and Pyrotechnics Sectional Committee, CHD 26

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FOREWORD

(*Formal Clause to be added later*)

Ammonium nitrate is extensively used both in commercial and military explosives. Small crystals of ammonium nitrate with a large surface-volume ratio, facilitates the manufacture of amatol.

This standard was first published in 1967. The original standard covered only one grade of ammonium nitrate. In the first revision, four grades of ammonium nitrate depending upon the use, were specified namely, for ammonium nitrate fuel oil (ANFO), slurry explosives, nitroglycerine-based explosives and defence.

In this revision, instrumental test methods for the determination of iron, lead, copper, zinc, manganese, chloride and sulphate have been added as alternate test methods.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard

AMMONIUM NITRATE FOR EXPLOSIVES — SPECIFICATION

(*Second Revision*)

1 SCOPE

This standard prescribes requirements and the methods of sampling and test for ammonium nitrate intended primarily for use in explosives.

2 REFERENCE

The Indian Standards listed in **Annex A** contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

3 GRADES

The material shall be of the following four grades namely:

a) *Grade* 1 — For ammonium nitrate fuel oil (ANFO);
b) *Grade* 2 — For slurry type explosives;
c) *Grade* 3 — For nitroglycerine-based explosives; and
d) *Grade* 4 — For defence purposes.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of prills, lumps or crystals, as agreed to between the purchaser and the supplier, and colourless or showing only a brownish tint. It shall be free from grit, foreign matter and visible impurities.

4.1.1 The different grades of material shall be of the following sizes:

a) *Grade* 1 — 0.8 mm to 2.8 mm;
b) *Grade* 2 — 2.9 mm to 100 mm; and
c) *Grade* 3 and 4 — as agreed to between the purchaser and the supplier.

4.2 The material may be with or without anti-caking agent of organic or inorganic nature, the limit for organic compounds being 0.1 percent. Where anti-caking agents are added they shall be compatible with the explosives and shall not cause undue frothing in the manufacturing process of explosives and the quantity and nature of the agent shall be declared by the supplier.

4.3 The material shall comply with the requirements specified in Table 1, when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col 7 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in polyethylene lined bags or in such other containers as agreed to between the purchaser and the supplier. The polyethylene sheet shall not be less than 0.05 mm thick.

5.2 Marking

Each package shall be legibly and indelibly marked with the following information:

a) Name and grade of the material;
b) Mass of the material in the package;
c) Manufacturers' name or his recognized trade-mark, if any;
d) Year of manufacture; and
e) Lot number to enable the batch of manufacture to be traced from records.

5.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Ammonium Nitrate for Explosives

(*Clause* 4.3)

Sl. No.	**Characteristic**	**Requirement for**				**Method of Test (Ref of Clause No. in Annex B)**
		Grade 1	Grade 2	Grade 3	Grade 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Moisture, percent by mass, *Max*	0.3	3.0	0.1	0.15	**B-2**
ii)	Matter insoluble in water, percent by mass, *Max*	0.3	0.05	0.01	0.25	**B-3**
iii)	Non-volatile matter, percent by mass, *Max*	0.2	—	0.1	1.0	**B-4**
iv)	Chlorides (as Cl), percent by mass, *Max*	—	0.05	0.006	0.05	**B-5 & B-23**
v)	Nitrates (as ammonium nitrate), percent by mass, *Max*	—	0.02	0.000 5	0.001	**B-6**
vi)	Acidity (as nitric acid), percent by mass, *Max*	—	0.01	0.01	0.01	**B-7**
vii)	Iron (as Fe), percent by mass, *Max*	—	0.002	0.002	—	**B-8 & B-24 & IS 13320**
viii)	Calcium (as calcium nitrite), percent by mass, *Max*	—	0.5	—	—	**B-9 & B-24**
ix)	*p*H of 10 percent aqueous solution	—	5 to 6	4 to 7	—	**B-10**
x)	Organic matter, percent by mass, *Max*	0.5	Nil	0.1	0.5	**B-11**
xi)	Grit, percent by mass, *Max*	—	Nil	Nil	Nil	**B-12**
xii)	Purity (on dry basis), percent by mass, *Min*	99.2	99.0	99.8	98.7	**B-13**
xiii)	Metals and compounds of metals other than alkali metals, calcium and iron, percent by mass, *Max*	—	—	0.02	0.02	**B-14 & B-24**
xiv)	Phenols or phenolic bodies	—	—	To Pass the test	To Pass the test	**B-15**
xv)	Pyridine, percent by mass, *Max*	—	—	Nil	0.003	**B-16**
xvi)	Thiocyanate (as ammonium thiocyanate), percent by mass, *Max*	—	—	Nil	0.002	**B-17**
xvii)	Prussian blue and allied compounds (as Prussian blue), percent by mass, *Max*	—	—	Nil	0.1	**B-18**
xviii)	Oil absorption, percent by mass, *Min*	7	—	—	—	**B-19**

xix)	Blue Density g/cm^2	0.75 to 0.85	—	0.92 to 1.02	—	**B-20**
xx)	Total nitrogen, percent by mass, *Min*	34.5	—	—	—	**B-21**
xxi)	Sulphates (as ammonium sulphate) percent by mass, *Max*	—	—	0.03	0.6	**B-22 & B-23**

6 SAMPLING

The procedure for drawing representative samples from a lot, the number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex C.

Annex A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*IS Title*
IS 170 : 2020	Acetone — Specification (*fifth revision*)
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 887 : 1977	Specification for animal tallow (*second revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision)*
IS 1460 : 2025	Automotive diesel fuel — Specification (*seventh revision*)
IS 2362 : 1993	Determination of water by karl fischer method — Test method (*second revision*)
IS 3025 (Part 2) : 2019 ISO 11885 : 2007	Methods of sampling and test (physical and chemical) for water and wastewater Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP – OES) (*first revision*)
IS 3025 (Part 65) : 2025 ISO 17294-2 : 2023	Methods of sampling and test physical and chemical for water and wastewater Part 65 Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*second revision*)
IS 13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method

ANNEX B

(*Clause* 4.3)

ANALYSIS OF AMMONIUM NITRATE FOR EXPLOSIVES

B-1 QUALITY OF REAGENTS

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

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

B-2 DETERMINATION OF MOISTURE

B-2.1 Two methods are prescribed, namely, air-oven method and Karl Fisher method. In case of dispute, Karl Fisher method shall be the referee method.

B-2.2 Air-Oven Method

B-2.2.1 *Procedure*

Weigh rapidly 10 g of the material and transfer it to a weighing dish with a close-fitting lid. Close the dish and weigh it accurately. Lift the lid and expose the material in an oven at 100 °C ± 2 °C for 2 h. Then close the lid, remove the dish to a desiccator for about half an hour to cool and reweigh it. Repeat the operation till a constant mass is obtained.

B-2.2.2 *Calculation*

$$\text{Moisture, percent by mass} = 100 \times \frac{M_1 - M_2}{M_1 - M}$$

where,

M_1 = mass of the material and the dish before heating, in g;

M_2 = mass of the material and the dish after heating in g; and

M = mass of empty dish, in g.

B-2.3 Karl Fisher Method

B-2.3.1 Carry the determination according to the method prescribed in IS 2362.

B-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

B-3.1 Procedure

Take 100 g of the sample in a 500 ml beaker and dissolve in about 200 ml of hot water. Filter the residue through Whatman No. 40 filter paper. Wash the residue with water till free from all soluble matter. Dry in an oven maintained at 100 °C ± 2 °C. Brush out the residue in a weighed silica crucible and weigh. Deduct the quantity of the declared anti-caking agent insoluble in water, if any, from the dried residue.

B-3.1.1 *Calculation*

$$\text{Matter insoluble in water, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass of the dried residue, in g; and

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

B-4 DETERMINATION OF NON-VOLATILE MATTER

B-4.1 Procedure

Heat a porcelain or silica basin with cover about 6 cm in diameter in an oven at 100 °C ± 2 °C for half an hour. Cool it in a desiccator and weigh. Place about 10 g of the material in the dish, moisten the ammonium nitrate with sulphuric acid, replace the cover and weigh accurately. Uncover the dish and heat the sample initially over low flame till the fume subsides and finally over strong flame having a temperature of 800 °C ± 20 °C for 1 h. Remove the burner and replace the cover, cool the basin and the cover in a desiccator to room temperature and weigh. Repeat the operation till constant mass is obtained.

B-4.2 *Calculation*

Calculate the non-volatile matter as follows:

$$\text{Non} - \text{volatile matter, percent by mass} = 100 \times \left(\frac{M_3 - M_1}{M_2 - M_1}\right)$$

where,

M_1 = mass of the empty dish and the cover, in g;

M_2 = mass of the dish and the cover with the sample taken, in g; and

M_3 = mass of the dish and the cover with the sample after heating, in g.

B-5 DETERMINATION OF CHLORIDES

B-5.1 Outline of the Method

Chloride is completely precipitated as silver chloride by the addition of an excess of standard silver nitrate solution and the excess is back titrated with ammonium thiocyanate.

B-5.2 Reagents

B-5.2.1 *Silver Nitrate Solution* — 0.1 N

B-5.2.2 *Ammonium Thiocyanate* — 0.1 N

B-5.2.3 *Concentrated Nitric Acid*

B-5.2.4 *Ferric Alum Indicator* — 40 percent

B-5.2.5 *Nitrobenzene*

B-5.3 Procedure

Weigh about 10 g of the sample accurately in a 250 ml conical flask. Dissolve with heating in 100 ml of water. Add 5 ml of concentrated nitric acid followed by 25 ml silver nitrate solution through a pipette. Shake for two minutes and titrate against 0.1 N thiocyanate using 2 ml of ferric alum indicator and 5 ml of nitrobenzene till there is a persistent colour change. Run a blank with all the reagents and water but without the sample.

B-5.4 Calculation

$$\text{Chloride, percent by mass} = (A - B) \times N \times \frac{35.45}{1\,000} \times \frac{100}{M}$$

where,

A = volume of ammonium thiocyanate consumed for blank titration;

B = volume of ammonium thiocyanate consumed for sample titration;

N = normality of ammonium thiocyanate solution; and

M = mass of sample taken for the test, in g.

B-5.5 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **B-23**.

B-6 DETERMINATION OF NITRITE

B-6.1 Two methods are prescribed, namely, colorimetric method and volumetric method. Colorimetric method is recommended for routine analysis. In case of dispute, volumetric method shall be the referee method.

B-6.2 Colorimetric Method

B-6.2.1 *Outline of the Method*

When dilute solutions of sulphanilic and α-naphthylamine are acted upon by nitrous acid, a red colouration is produced which may be used for the colorimetric determination of nitrites. The full colour does not appear at once. A satisfactory colour comparison can be made after 10 to 15 minutes provided the sample and standard are treated in the same manner.

B-6.2.1 *Reagents*

B-6.2.1.1 *Sulphanilic acid reagent* (*solution* 1)

Dissolve 0.6 g of sulphanilic acid in 100 ml of 20 percent (*v/v*) dilute hydrochloric acid.

B-6.2.1.2 *α-Naphthylamine reagent* (*solution* 2)

Dissolve 0.48 g of α-naphthylamine in 100 ml of 20 percent (*v/v*) dilute hydrochloric acid.

B-6.2.1.3 *Standard nitrite solution*

Dissolve 4.93 g of sodium nitrite in water and dilute with deionized water. Dilute 10 ml of standard solution to one litre with deionized water.

B-6.2.1.4 *Sodium acetate* (2 *M*)

Dissolve 16.4 g of anhydrous sodium acetate in water and dilute to 100 ml with water.

B-6.2.1 *Procedure*

Place the quantity of the sample containing about 0.03 mg or less of nitrite in a 50 ml volumetric flask. Add 1 ml of sulphanilic acid reagent, mix well and allow to stand at least for 3 minutes but not more than 10 minutes at room temperature in diffused light. Introduce 1 ml of α-naphthylamine reagent and 1 ml of 2 M sodium acetate solution to act as buffer (*p*H 2 to 2.5). Dilute to volume and mix well. Use a photoelectric colorimeter with a green filter and measure transmittance. Construct a reference curve with known nitrate standards, preferably in the nitrite-nitrogen range of the sample and measure the concentration of the nitrite in the sample solution from the reference curve.

B-6.2.2 *Calculation*

The concentration of nitrite is directly read from the reference curve and calculated as ammonium nitrite.

B-6.3 Volumetric Method

B-6.3.1 *Reagents*

B-6.3.1.1 *Potassium permanganate solution* — N/50.

B-6.3.1.2 *Sulphuric acid* — (1:1).

B-6.3.2 *Procedure*

B-6.3.2.1 Weigh accurately about 30 g of sample and transfer to a 100 ml volumetric flask and dissolve with shaking in 40 ml of water. Make up the volume to the mark with water. Stopper the flask and shake well. Take up the solution in a burette.

B-6.3.2.2 Take 2.5 ml of freshly prepared N/50 potassium permanganate solution in a conical flask. Add 5 ml of sulphuric acid (1: 1) Dilute to 40 ml with water and heat to around 60 °C and titrate against sample solution taken in the burette till the pink colour disappears.

B-6.3.3 *Calculation*

1 ml of 1 N potassium permanganate = 0.032 0 g of ammonium nitrite.

B-7 DETERMINATION OF ACIDITY AS NITRIC ACID

B-7.1 Outline of the Method

The dried material is dissolved in neutral acetone, water added to the solution and then titrated with sodium hydroxide solution.

B-7.2 Reagents

B-7.2.1 *Acetone* — Neutralized to methyl red indicator (*see* IS 170)

B-7.2.2 *Standard Sodium Hydroxide Solution* — 0.01 N.

B-7.2.3 *Methyl Red Indicator*

Dissolve 1 g of methyl red (water soluble) in water and dilute the solution to 1 litre.

B-7.3 Procedure

Add about 2 g to 10 g of the dried material weighed accurately to 0.01 g, to 100 ml of acetone. Shake the mixture vigorously for several minutes until solution is complete. Add 100 ml of water to solution with constant stirring. Boil off the acetone on a water bath, cool, filter through a filter paper (Whatman No. 1 or its equivalent), wash with two 10 ml portions of water and titrate the filterate with standard sodium hydroxide solution using methyl red as indicator.

B-7.3.1 Run a blank determination using the same quantities of reagents.

A-*Calculation*

$$\text{Acidity (as nitric acid), percent by mass } = \frac{6.3\ (V_2 - V_1)N}{M}$$

where,

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

V_2 = volume in ml of standard sodium hydroxide solution required by the material,

N = normality of standard sodium hydroxide solution, and

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

B-8 DETERMINATION OF IRON

B-8.1 Outline of the Method

Ferric (but not ferrous) iron reacts with thiocyanate to give a series of intensely red coloured complexes which remain in true solution. In the colorimetric determination, excess of thiocyanate should be used since this increases the intensity and also stability of the colour. Strong acid (hydrochloric or nitric acid concentration 0.052 M to 0.05 M) should be present to suppress the hydrolysis of ferric iron.

B-8.2 Reagents

B-8.2.1 *Potassium Thiocyanate Solution* — Dissolve 20 g of potassium thiocyanate in 100 ml of water.

B-8.2.2 *Concentrated Nitric Acid — see* IS 264.

B-8.2.3 *Standard Solution of Iron* (*Ferric*)

Dissolve 0.864 g of ferric ammonium sulphate in water, add 10 ml of concentrated nitric acid and dilute it to one litre. One ml of this solution is equivalent to 0.1 mg of iron (as Fe).

B-8.3 Procedure

Dissolve accurately 5 g of the material in 1: 1 water nitric acid mixture and evaporate to nearly dryness to expel excess of acid. Dilute slightly with water, oxidize any ferrous ion to the ferric state with dilute potassium permanganate solution (2g/l), adding slowly dropwise until a slight pink colour remains after stirring well. Make up the solution to 250 ml or other suitable volume. Place 50 ml of the solution in a Nessler cylinder, add 5 ml of thiocyanate solution and 2 to 4 ml of 4 N nitric acid. Add the same amounts of reagents to 50 ml of water contained in a similar Nessler cylinder and run in standard iron solution from burette (use long glass rod with flattened end for mixing) until colours are matched. Note the exact volume (X ml) of the standard iron solution added. Repeat determination using (50 – X) ml of water. Comparison of the standard and unknown should be made soon after preparation since the colour may fade on standing for a long time. For estimating the concentration of ferric iron more accurately, the use of photoelectric colorimeter may be resorted to. A filter showing maximum transmission at or near 480 nm should be used. Standard solutions of ferric iron which are in the range of the concentration of ferric iron likely to be present in the sample are prepared and the transmittance of each such solution measured with photoelectric colorimeter. A reference curve is then prepared by plotting the concentration versus transmittance. Now the transmittance of the sample solution is also measured and from the reference curve, the concentration of ferric iron as [$Fe(NO_3)_3$] is directly read.

B-8.4 Calculation

$$\text{Ferric iron [as } Fe(NO_3)_3 \text{ percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of ferric iron [as $Fe(NO_3)_3$] as found out from the reference curve, and

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

B-8.5 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at B-24 or ICP-MS method as prescribed in IS 3025 (Part 65) or AAS method as prescribed in IS 13320.

B-9 DETERMINATION OF CALCIUM

B-9.1 Outline of the Method

Calcium, like some other metal ions, is complexed by disodium dihydrogen ethylene diamine tetra-acetate (EDTA) by choosing the proper *p*H and indicator. Using calcon as indicator, calcium can be estimated in the presence of magnesium since magnesium is precipitated quantitatively as magnesium hydroxide in the *p*H range of the reaction.

B-9.2 Reagents

B-9.2.1 *Diethylamine*

B-9.2.2 *Calcon Indicator* — Prepared by dissolving 0.2 g of the dyestuff in 50 ml of methyl alcohol.

B-9.2.3 *Standard Disodium Dihydrogen Ethylenediamine Tetra Acetate* (*EDTA*) *Solution*

Weigh 3.772 5 g of EDTA, dissolve in water free from polyvalent ions (distilled water for this purpose should be passed through a column of cation exchange resins in the sodium form) and make up to a volume of 1 000 ml. Concentration of the resulting solution will be 0.01 M. (If the EDTA is not of analytical reagent grade, then the solution prepared needs to be standardised against standard zinc chloride solution or magnesium chloride solution which is prepared from analytical reagent grade material or corresponding metal pellets of analytical reagent grade).

B-9.2.4 *Sample Solution*

Weigh about one gram of the material accurately, dissolve in water from free polyvalent ions, filter and make up to 100 ml.

B-9.3 Procedure

Pipette out 10 ml of the sample solution (*see* **B-9.2.4**) into 250 ml conical flask. Add about 40 ml of water and 5 ml of diethylamine, giving the mixture a *p*H of about 12.5. Under these conditions, magnesium, if present, precipitates quantitatively as the hydroxide. Add four drops of calcon indicator to the solution and titrate with standard EDTA solution with shaking until the colour changes from pink to a pure blue.

B-9.4 Calculation

$$\text{Calcium [as Ca (NO}_3)_2\text{] percent by mass } = \frac{1.64\ \times \text{VM}}{\text{A}}$$

where

V = volume in ml of standard EDTA solution,

M = molarity of standard EDTA solution, and

A = mass in g of material taken for the test

B-9.5 Alternative Method

Calcium may alternatively be determined by ICP-OES instrumental test method as prescribed at B-24 or ICP-MS method as prescribed in IS 3025 (Part 65).

B-10 DETERMINATION OF *p*H

B-10.1 Procedure

Dissolve 10.0 g of the material in 100 ml of water and measure the *p*H of the solution by means of a suitable *p*H meter using glass electrodes.

B-11 DETERMINATION OF ORGANIC MATTER

B-11.1 Procedure

Weigh about 50 g of the sample accurately in a tall sintered-glass grade 2 thimble with a small hole at the top of wall for tying a small piece of wire. Put the thimble gently in the soxhlet extraction chamber with the help of the wire. Take 200 ml of the suitable solvent (dichloromethane, diethyl ether, acetone, N-butyl acetate, etc.) in the flask containing a few glass beads. Fit the apparatus on a boiling water bath and continue the extraction for three hours. Remove the condenser and take out the thimble. Replace the condenser and continue boiling till majority of the solvent is recovered in the flask to a tared flat bottom glass basin with cover. Rinse the flask twice with the suitable solvent and transfer in the basin. Put the basin in a boiling water bath at 95 °C ± 2 °C for two hours.

At the end of this period, replace the cover, cool the basin and the cover in a desiccator to room temperature and weigh. Repeat the drying operation till constant mass is obtained.

NOTE — When required, the manufacturers shall inform about the name of the solvent to be used.

B-11.2 Calculation

$$\text{Organic matter, percent by mass } = \frac{\mathrm{M_2}}{\mathrm{M_1}} \times 100$$

where,

M_2 = mass in g of the residue after complete evaporation of the solvent extract, and

M_1 = mass in g of the material taken for test.

B-12 DETERMINATION OF GRIT

B-12.1 Procedure

Weigh about 50 g of the material into a 500 ml beaker, dissolve in adequate quantity of water and allow to stand until the insoluble material settles. Decant the supernatent liquor and filter the rest using a filter paper. Ignite the filter paper with the residue in a muffle furnace. Boil the residue for about 30 minutes with 20 ml aqua regia. After cooling the mixture, drain off the aqua regia and wash the residue thoroughly with water. Dry the residue in an air oven and sieve it through 125 μm IS-sieve. Weigh the portion retained on the sieve and test for soda glass scratching. If it scratches soda glass then report it as percent grit.

B-12.2 Calculation

$$\text{Grit, percent by mass} = \frac{M \ \times 100}{M_1}$$

where,

M = mass in g of the residue retained on 125 μm IS-sieve, and

M_1 = mass in g of the material taken for test.

B-13 DETERMINATION OF PURITY ON DRY BASIS

B-13.1 Outline of the Method

Ammonia from ammonium nitrate reacts quantitatively with excess of formaldehyde to produce nitric acid.

$$4NH_4NO_3 + 6HCHO \rightleftarrows (CH_2)_5N_4 + 4HNO_3 + 6H_2O$$

This nitric acid can be titrated against standard alkali

B-13.1 Reagents

B-13.1.1 *Sodium Hydroxide* — 0.2 N.

B-13.1.2 *Hydrochloric Acid* — 0.2 N.

B-13.1.3 *Phenolphthaline* — 1 percent in alcohol.

B-13.1.4 *Formaldehyde 1:1*

Mix equal volumes of AR grade formaldehyde (37 percent *m/m*) and water. Add few drops of phenolphthaline indicator and titrate against standard alkali to its light pink end point.

B-13.2 Procedure

Weigh exactly 0.4 g of sample into a 250 ml conical flask and dissolve in 20 ml of water. Add 50 ml of formaldehyde 1 : 1 reagent and warm the flask. Cool to room temperature and titrate against standard alkali till permanent light pink colour appears. Drain another 2 ml of alkali and note down the volume (V_1). Keep it for 10 minutes and titrate against standard hydrochloric acid using a microburette till the solution becomes colourless and note down the volume (V_2). Repeat the same experiment till concordant values are obtained.

B-13.3 Calculation

$$\text{Volume of sodium hydroxide required (V)} = \mathrm{V_1} - \frac{(\mathrm{V_2} \times \mathrm{N_2})}{\mathrm{N_1}}$$

$$\text{Percent purity of ammonium nitrate } \frac{\mathrm{V} \times \mathrm{N_1} \times 8.005}{\mathrm{M}}$$

$$\text{Purity (on dry basis), percent } = \frac{100 \times \text{purity as obtained above}}{(100 - \text{moisture percent})}$$

where

N_1 = normality of sodium hydroxide,

N_2 = normality of hydrochloric acid, and

M = mass of the sample, in g.

B-14 DETERMINATION OF METALS AND COMPOUNDS OF METALS OTHER THAN ALKALI METALS, CALCIUM AND IRON

B-14.1 Procedure

Treat 5 g of the material in a porcelain crucible with 1 ml of concentrated sulphuric acid. Drive off the excess acid by gentle heating and heat the residue strongly. Extract the residue with dilute hydrochloric acid and test qualitatively for the presence of metallic radicals (other than alkali metals, iron and calcium) like lead, copper zinc and manganese. If any metallic impurity is detected, estimate quantitatively by any standard colorimetric or instrumental method.

B-14.2 Alternative Method

Lead, Copper, Zinc and Manganese may alternatively be determined by ICP-OES instrumental test method as prescribed at B-24 or ICP-MS method as prescribed in IS 3025 (Part 65).

B-15 DETERMINATION OF PHENOL OR PHENOLIC BODIES

B-15.1 Procedure

To a solution of 10 g of the material in 15 ml of water, add 100 ml of bromine water and 1 ml of concentrated hydrochloric acid. After vigorous shaking, no precipitate should appear in the solution after keeping for one hour.

B-16 DETERMINATION OF PYRIDINE

B-16.1 Triturate a little powdered borax rapidly with about 5 g of the material in a mortar. If the odour of pyridine is detected, estimate the amount of pyridine present as follows.

B-16.2 Procedure

Dissolve 100 g of the material in 300 ml of water in a 500 ml flask and add a few drops of methyl orange solution. Fit the flask with a rubber stopper through which passes a tap-funnel and a glass outlet tube. The outlet tube incorporating a bulb or other device to guard against a suck back, leads into a 500 ml conical flask containing sodium hydroxide solution and 4 ml of bromine, and dips below the surface of this solution. Close the conical flask with a rubber stopper through which also passes a distilling trap attached to a water condenser. Attach to the exit tube of the condenser a 250 ml conical flask as receiver containing a little water and fitted with guard tube of moistened glass. beads.

B-16.3 Add 5 ml of 1 N sodium hydroxide solution to the ammonium nitrate solution through the tap funnel and distil the solution slowly into the hypobromite solution. When about 50 ml of the ammonium nitrate solution has been distilled, steam distil the hypobromite solution by warming the flask of hypobromite and continuing the distillation of the ammonium nitrate solution. Titrate the pyridine so collected in the receiver with 0.1 N hydrochloric acid using methyl orange as indicator.

B-16.4 Calculate the factor to be used in calculating the amount of pyridine present from the results of titration of aqueous solutions, containing known quantities of pyridine.

B-17 DETERMINATION OF THIOCYANATE

B-17.1 Procedure

Dissolve 5 g of the material in about 80 ml of water. Filter the solution, if necessary, and make it up to 100 ml in a Nessler cylinder. Prepare similarly a series of standard solutions containing 5 g of pure thiocyanate free ammonium nitrate and different known amounts of ammonium thiocyanate. Add 2 ml of 10 percent ferric alum solution (containing 50 ml of concentrated nitric acid/litre) to the contents of each cylinder, with stirring. Determine the amount of thiocyanate present in the material by comparing the colour of the solution under test with the standard solutions.

B-18 DETERMINATION OF PRUSSIAN BLUE AND ALLIED PRODUCTS

B-18.1 Procedure

Warm 10 g, approximately, of the material with about 20 ml of 1 N nitric acid solution. If the resulting solution is coloured blue or contains blue coloured particles, estimate the Prussian blue as follows.

B-18.1.1 Dissolve 100 g of the material in about 100 ml of water and acidify the solution with 20 ml of 1 N nitric acid. Heat for 5 min and then filter. Treat the residue of prussian blue on the filter paper with 100 ml of 1 N potassium hydroxide solution, added in consecutive small portions and make up the resulting filtrate, containing potassium ferrocyanide, to a volume of 200 ml.

B-18.1.2 Transfer a suitable aliquot portion of the ferrocyanide solution to a Nessler cylinder and add a 2 ml excess of 1 N nitric acid. Dilute the solution to 100 ml and add 5 ml of 10 percent ferric alum solution with stirring.

B-18.1.3 Prepare similarly a series of standard solutions in Nessler cylinders, containing the same quantities of alkali, acid and ferric alum as in the first Nessler cylinder but with different known quantities of potassium ferrocyanide.

B-18.2 Determine the amount of prussian blue present in the material by comparing the colour of the solution under test with the standard solutions.

B-19 DETERMINATION OF OIL ABSORPTION

B-19.1 Procedure

10 g of sample to be tested is placed in a dry 50 ml beaker. Ten ml of diesel oil (HSD conforming to IS 1460 is added to the sample in the beaker and the contents are left for exactly 10 minutes. The contents of the beaker are then transferred to a porcelain buchner funnel (6 cm internal diameter) equipped with a Whatman No. 1 filter paper. A suction of 150 mm mercury is applied for three minutes. The oiled sample is then transferred to a previously weighed watch glass. The mass of the oiled sample is calculated from the combined masses of the sample and the watch glass. The percent oil absorption is calculated as follows:

B-19.2 Calculation

$$\text{Oil absorption, percent by mass} = \frac{M_2 - M_1}{M_1} \times 100$$

where,

M_2 = mass in g of oiled sample, and

M_1 = mass in g of original sample

B-20 DETERMINATION OF BULK DENSITY

B-20.1 Apparatus

B-20.1.1 Assemble the apparatus as shown in Fig. 1. The measuring cylinder *A* shall be of 20 ml capacity and shall conform to IS 878. The base of the measuring cylinder shall be ground flat. The distance between the flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 25 mm ± 2 mm.

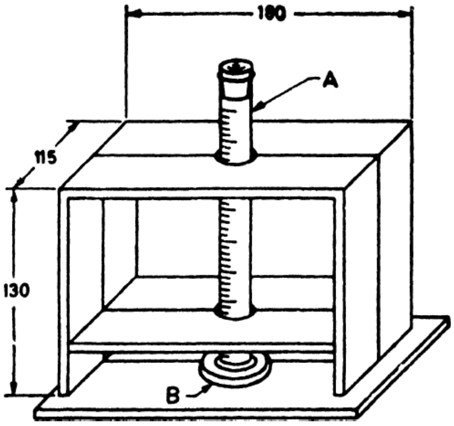

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

B-20.1.2 The rubber base pad *B* shall have a shore hardness of 42 to 50.

B-20.1.3 Pans of the balance hall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

B-20.2 Procedure

Place 50 g of the material in the graduated cylinder fitted in the apparatus and tap it until a constant volume of the material is obtained. Note down the volume of the material

B-20.3 Calculation

$$\text{Bulk density, g/cm}^3 = \frac{\text{M}}{\text{V}}$$

where

M = mass in g of the material taken for the test, and

V = volume of the material in the cylinder.

B-21 DETERMINATION OF TOTAL NITROGEN (AS N)

B-21.1 Outline of the Method

Ammonia is distilled off from an alkaline solution and absorbed in standard hydrochloric acid. The excess of acid is titrated with standard hydroxide solution.

B-21.2 Apparatus — Ammonia distillation apparatus as shown in Fig. 2.

B-21.3 Reagents

B-21.3.1 *Sodium Hydroxide Solution* — 30 percent (*m/v*).

B-21.3.2 *Standard Hydrochloric Acid* — 0.5 N.

B-21.3.3 *Standard Sodium Hydroxide Solution* — 0.5 N.

B-21.3.4 *Mixed Methyl Red Indicator Solution*

Dissolve 0.1 g of methyl red in 50 ml of ethyl alcohol and 0.05 g of methylene blue in 50 ml of ethyl alcohol and mix the two solutions.

B-21.3.5 *Devarda's Alloy* — Containing 45 parts of aluminium, 50 parts of copper and 5 parts of zinc.

B-21.4 Procedure

Weigh accurately 2.0 g of the prepared sample and transfer to the Kjeldahl flask, adding about 150 ml of water and a few porcelain beads. Add 6 g to 7 g Devarda's alloy. Connect the apparatus as shown in Fig. 2. Measure into the beaker exactly 125 ml of standard hydrochloric acid and add 4 drops to 5 drops of the mixed indicator solution. Place the beaker so that the end of the delivery tube is below the surface of the acid. Pour about 25 ml of sodium hydroxide solution (30 percent) into the tap funnel. Slowly drop the sodium hydroxide solution into the Kjeldahl flask by opening the tap of the funnel. Close the tap after delivering the entire quantity of sodium hydroxide. Start heating the Kjeldahl flask and circulate water in the condenser. After distilling about 150 ml into the receiver, remove the beaker and titrate the excess acid using 0.5 N sodium hydroxide solution. The end point is orange to green. Suggested indicator solution for the above determination is methyl red and bromocresol green solution. It is prepared by dissolving 0.1 g of methyl red in 50 ml of ethyl alcohol and mixing the two solution.

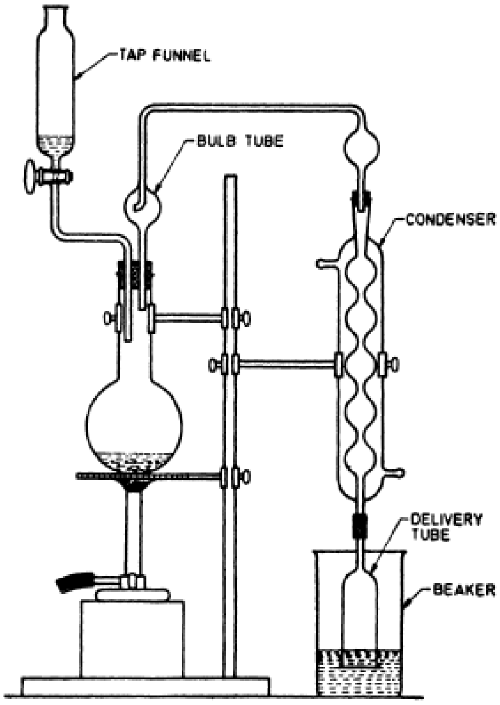

FIG. 2 APPARATUS FOR DETERMINATION OF NITROGEN

B-21.5 Calculation

$$\text{Total nitrogen (as N), percent by mass} = \frac{1.401 \times (V_1N_1 - V_2N_2)}{M}$$

where

V_1 = volume in ml of standard hydrochloric add used,

N_1 = normality of standard hydrochloric acid,

V_2 = volume in ml of standard sodium hydroxide, solution,

N_2 = normality of standard sodium hydroxide solution, and

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

B-22 DETERMINATION OF SULPHATES

B-22.1 Outline of the Method

Sulphates are determined by comparing the turbidity produced with barium sulphate solution, against a standard turbidity in Nessler cylinders.

B-22.2 Apparatus

B-22.2.1 *Nessler Cylinders* — 50 ml capacity.

B-22.3 Reagents

B-22.3.1 *Sodium Carbonate* — Solid.

B-22.3.2 *Dilute Hydrochloric Acid* — Approximately 2 N.

B-22.3.3 *Standard Sulphuric Acid* — 0.002 N.

B-22.3.4 *Barium Sulphate Reagent*

Dissolve 2.0 g of barium chloride in 75 ml of water, add 20 ml of 95 percent (*v/v*) ethyl alcohol and 5 ml of standard sulphuric acid. It shall be prepared afresh.

B-22.4 Procedure

Weigh accurately 10 g of the prepared sample into a porcelain dish and dissolve in 40 ml of water. Add 0.05 g of sodium carbonate and heat on a sand-bath at about 120 °C to volatilize water and then heat over a very low flame until the evolution of white fumes has ceased. Finally heat the dish at 600 °C in a furnace for 15 min. Cool, dissolve the residue in 10 ml of water, add 1 ml. of dilute hydrochloric acid and filter through a Whatman No. 41 filter paper. Transfer the filtrate to a Nessler cylinder, add 10 ml of water and 1 ml of barium sulphate reagent. Mix well and allow to stand for 5 min.

B-22.4.1 Prepare a series of sulphate standard turbidities with different known volumes of standard sulphuric acid and 1 ml of barium sulphate reagent in the similar manner and compare the turbidities. Calculate the sulphate content as ammonium sulphate in the sulphate standard in which the turbidity most closely matches with that in the solution of the material.

B-22.5 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at B-23.

B-23 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

B-23.1 Principle

B-23.1.1 Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger.

B-23.1.2 When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surfaces bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

B-23.1.3 Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

B-23.2 Equipment

B-23.2.1 *Anion Guard Column* — a protector of the separator column

B-23.2.2 *Anion Separator Column* — suitable for selective separation of ions under analysis.

B-23.2.3 *Anion Suppressor device*

Anion micro membrane suppressor is used to analyse the data

B-23.2.4 *Detector* — conductivity detector

B-23.2.5 *Software*

Software suitable for control of various operating parameters, receiving inputs and analysis of all data

B-23.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

B-23.3 Reagents

B-23.3.1 Glass or Polyethylene Sample bottles.

B-23.3.2 Distilled Water or Deionized Water free from the Anions of interest.

B-23.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litre of water.

B-23.3.4 *Micro Membrane Suppressor Solution* — (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

B-23.4 Standard solutions

B-23.4.1 *Chloride*

Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water.

B-23.4.2 *Sulphate*

Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

B-23.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate are responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

B-23.6 Procedure

B-23.6.1 Dissolve between 1 g to 5 g samples in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample.

B-23.6.2 Use the loop of same size for the standards and samples. Record the peak in size and area units. An automate constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used-

B-23.6.3 Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

B-23.7 Data analysis and Calculations

B-23.7.1 Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

B-23.7.2 Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

B-24 DETERMINATION OF LEAD, CALCIUM, COPPER, IRON, ZINC AND MANGANESE BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-24.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

B-24.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences

B-24.2.1 Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

B-24.2.2 Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(Clauses B-24.2.1, B-24.2.2 and B-24.4)

Sl. No.	**Element**	**Wavelength (nm)**	**Approximately Achievable limits**		**Interfering Elements**
			Radial viewing (µg)	**Axial viewing (µg)**	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iv)	Cu	324.754	9	2	Cr, Fe, Mo, Ti
		327.396	4	3	Co, Ti
v)	Zn	202.548	-	(3)	Cr, Cu, Co, Ni
		206.200	13	5	Cr
		213.857	3.3	1	Cu, Fe, Ni
vi)	Mn	257.610	1	0.4	Cr, Fe, Mo, W
		293.305	(20)	8	Al, Cr, Fe, Ti

B-24.3 Reagents and Solutions

B-24.3.1 *Nitric Acid (65 percent) Suprapure*

B-24.3.2 *Standard Stock Solution*

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of Lead, Iron, calcium, manganese, copper zinc in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-24.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml, and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml, and 0.5 µg/ml solution of respective elements under reference.

B-24.3.4 *Sample Preparation*

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with distilled water.

NOTE — Sample should be clear before injecting to the instrument.

B-24.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

B-24.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsenic generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

B-24.5 Procedure

B-24.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (B-24.3.3). The relationship between concentration and intensity is linear up

to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

B-24.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration of elements in µg/ml.

NOTE — It is recommended that IS 3025 (Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis

B-24.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements

and establish a calibration plot.

ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

ANNEX C

(*Clause* 6)

SAMPLING OF AMMONIUM NITRATE FOR EXPLOSIVES

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.2 Samples shall not be drawn in an exposed place.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

C-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

C-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.7 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* **5.2**) and the date of sampling.

C-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

C-2 SCALE OF SAMPLING

C-2.1 Lot

In a single consignment of the material all the containers of the same, type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch type and size shall be grouped together and each such group shall constitute a separate lot.

C-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 3.

Table 3 Scale of Sampling

(*Clause* **C-2.1.1**)

Sl. No. (1)	**LOT Size** (N) (2)	**Sample Size** (n) (3)
i)	Up to 15	3
ii)	16 to 25	4
iii)	26 to 50	5
iv)	51 to 100	7
v)	101 and above	10

C-2.1.2 In order to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure is recommended for use:

Arrange the containers in the lot in a systematic order and starting from anyone, count them as 1, 2, 3, , etc, up to *r* and so on, where *r* is the integral part of *N/n* (*N* and *n* being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.

C-3 PREPARATION OF TEST SAMPLES

C-3.1 From each of the containers selected, draw a small representative portion of the material not less than 100 g in mass.

C-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly, to form a composite sample of mass not less than 150 g. The composite sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

C-3.3 The remaining portion of the material from each container shall be divided into 3 equal parts and transferred to separate bottles, giving full identification particulars of the samples on the bottle. The material in each bottle constitutes an individual sample. One of these three sets (each set containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

C-3.4 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for the determination of moisture shall be performed on each of the individual samples.

C-4.2 Tests for the determination of all the remaining characteristics given in 3 and Table 1 shall be carried out on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 From each set of the individual test results for moisture, the average ($\bar{X}$) and the range (R) of the test results shall be computed (range R is defined as the difference between the maximum and the minimum values of the test results).

C-5.2 For declaring the conformity of the lot to the specification requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.