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# *Indian Standard*

SPECIFICATION FOR UREA, TECHNICAL

*( First Revision )*

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# Indian Standard

## SPECIFICATION FOR UREA, TECHNICAL

### ( First Revision )

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# *Indian Standard*

## SPECIFICATION FOR UREA, TECHNICAL ( *First Revision* )

### 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 14 July 1975, after the draft finalized by the Acids and Fertilizers Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was first published in 1961 when it covered the industrial and the fertilizer grades. Later, it was decided to prepare a separate standard for fertilizer grade urea ( published as IS : 5406-1969 ). In the present revision covering technical grade urea, the biuret content has been lowered since it was seen that manufacturers of technical grade urea were able to comply with the reduced limit and since the consumers wanted as low a limit as possible. Additional requirements have been prescribed for ammonium salts, residue on ignition, melting point, alkalinity value and buffer value.

**0.3** The revision has been prepared taking into consideration the needs of the plastics and adhesives industries.

**0.4** The International Organization for Standardization has brought out the following standards on urea for industrial use, and corresponding portions in this standard agree substantially with them:

- ISO/R 1592-1970 Urea for industrial use. Determination of nitrogen. Volumetric method.
- ISO/R 1594-1970 Urea for industrial use. Determination of ash. Gravimetric method.
- ISO/R 1595-1970 Urea for industrial use. Determination of iron content 2, 2' — Bipyridyl photometric method.
- ISO 2749-1973 Urea for industrial use. Measurement of the pH of a solution of urea of conventional concentration ( 100 g/l ) — Potentiometric method.
- ISO 2751-1973 Urea for industrial use. Determination of the buffer coefficient — Potentiometric method.
- ISO 2754-1973 Urea for industrial use. Determination of biuret content — Photometric method.

## IS:1781-1975

0.5 For the determination of particle size, IS Sieves (conforming to IS: 460-1962\*) have been prescribed. Where IS Sieves are not available, equivalent test sieves as judged from aperture size may be used.

0.6 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: 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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### 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for urea, technical. It does not cover the material used as fertilizer.

### 2. REQUIREMENTS

2.1 **Description** — The material shall be in the form of white prills or crystals. It shall be free from visible impurities and from dust.

2.2 In the form of prills, the material shall pass through 2.00 mm IS Sieve and not less than 80 percent by mass of it shall be retained on 0.85 mm IS Sieve.

2.3 The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of the table.

2.4 **Optional Requirements** — Subject to agreement between the purchaser and the supplier, the material shall also conform to the following requirements when tested according to the method given in A-10:

- a) Alkalinity value — 2.0 *Max*, and
- b) Buffer value — 4.0 *Max*

### 3. PACKING AND MARKING

3.1 **Packing** — The material shall be packed in polyethylene lined jute bags or in such other containers as agreed to between the purchaser and the supplier.

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\*Specification for test sieves (*revised*).

†Rules for rounding off numerical values (*revised*).



**TABLE 1 REQUIREMENTS FOR UREA, TECHNICAL**  
( Clause 2.3 )

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST ( REF TO CL No. IN APPENDIX A )
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, <i>Max</i>	0.5	A-3
ii)	Nitrogen, percent by mass ( <i>on dry basis</i> ), <i>Min</i>	46.0	A-4
iii)	Biuret, percent by mass, <i>Max</i>	0.8	A-5
iv)	Free ammonia ( as $\text{NH}_3$ ), percent by mass, <i>Max</i>	0.015	A-6
v)	Ammonium salts ( as $\text{NH}_3$ ), percent by mass, <i>Max</i>	0.01	A-7
vi)	Residue on ignition, percent by mass, <i>Max</i>	0.002	A-8
vii)	Iron ( as Fe ), percent by mass, <i>Max</i>	0.000 2	A-9
viii)	pH of 10 percent solution	7.0 to 9.5	A-10
ix)	Melting point, °C	131 to 133	A-11

**3.2 Marking** — The containers shall be securely closed and marked with the following information:

- a) Name of the material;
- b) Name of the manufacturer;
- c) Mass in kg of the material in the container; and
- d) Recognized trade-mark, if any.

**3.2.1** The containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn as prescribed in Appendix B.

## APPENDIX A

( Clause 2.3 )

### METHODS OF TEST FOR UREA, TECHNICAL

#### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1960\* ), shall be employed in tests.

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

#### A-2. PREPARED SAMPLE

**A-2.1** Mix the sample well by rotating the bottle several times and transfer a portion immediately into a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Use this *prepared sample* for tests.

#### A-3. DETERMINATION OF MOISTURE

**A-3.1 Procedure** — Carry out the determination as prescribed in IS : 2362-1973†, using a weighed quantity of the *prepared sample* ( see A-2.1 ) containing 10 to 50 mg of water.

#### A-4. DETERMINATION OF NITROGEN

**A-4.0 General** — Nitrogen is determined by Kjeldahl method.

**A-4.1 Apparatus** — The apparatus, as assembled, is shown in Fig. 1. It consists of a round bottom Kjeldahl flask *A* of 500 ml capacity fitted with a two holed rubber stopper. Through one hole passes one end of the connecting bulb tube *B* and through the other hole passes the end of the dropping funnel *F*. The tube end of the bulb tube *B* is connected to the condenser *C* by a rubber stopper and the lower end of the condenser *C* is attached by means of a rubber tubing to a dip tube *D* which dips into a beaker *E* of 600 ml capacity.

#### A-4.2 Reagents

**A-4.2.1 Potassium Sulphate-Copper Sulphate Mixture** — Prepared by mixing 20 parts by mass of anhydrous potassium sulphate ( sodium sulphate may be used if potassium sulphate is not available ) and 1 part by mass of anhydrous copper sulphate.

**A-4.2.2 Concentrated Sulphuric Acid** — conforming to IS : 266-1961‡.

\*Specification for water, distilled quality ( revised ).

†Method for determination of water by Karl Fischer method ( first revision ).

‡Specification for sulphuric acid ( revised ).

**A-4.2.3 Standard Sulphuric Acid — 0.5 N.**

**A-4.2.4 Methyl Red-Methylene Blue Mixed Indicator** — Dissolve 0.1 g of methyl red in 50 ml of rectified spirit (conforming to IS: 323-1959\*) and add 0.05 g of methylene blue. When it has dissolved, dilute to 100 ml with the same rectified spirit.

**A-4.2.5 Sodium Hydroxide Solution** — approximately 45 percent (*m/v*).

**A-4.2.6 Standard Sodium Hydroxide Solution** — 0.5 N.

**A-4.3 Procedure** — Weigh accurately about 0.5 g of the *prepared sample* (see A-2.1) and transfer to the Kjeldahl flask *A*. Wash the neck of the flask with a little distilled water. Add 10 g of the potassium sulphate-copper sulphate mixture and 30 ml of concentrated sulphuric acid to the flask. Place the flask in an inclined position. Heat below boiling point until frothing ceases. Raise the temperature to bring the acid to brisk boiling. Continue the heating until the solution becomes straw-yellow in colour or practically water-white. Now remove the flask from the flame and cool. While cooling, carefully add about 250 ml of water, stirring during the addition of water. Add into the flask *A* a few drops of the mixed indicator and a few pieces of pumice stone to regulate boiling. Assemble the apparatus as shown in Fig. 1, with the tip of the condenser dipping in 50.0 ml of standard sulphuric acid in beaker *E* to which a few drops of the mixed indicator have been added.

Make sure that all connections are air-tight and water is flowing through the condenser. Pour through the dropping funnel sufficient sodium hydroxide solution to neutralize the solution and then 25 ml in excess. Then close the stop cock and shake the flask. Heat the flask and distil until about 250 ml of distillate has passed over. Detach flask *A* from the condenser and shut off the burner. Rinse the condenser thoroughly with water into the beaker *E*. Wash the dip tube *D* carefully so that all traces of the condensate are transferred to the beaker. When all the washings have drained into the beaker *E*, add two or three drops more of the mixed indicator and titrate with standard sodium hydroxide solution, stirring the solution thoroughly during titration.

**A-4.3.1** Carry out a blank using all reagents in the same quantities but without the material to be tested.

**A-4.4 Calculation**

$$\text{Nitrogen, percent by mass (on dry basis)} = \frac{70.04 (V_1 - V_2)}{M (100 - M_1)}$$

where

$V_1$  = volume in ml of standard (0.5 N) sodium hydroxide solution used to neutralize the acid in blank determination,

\*Specification for rectified spirit (*revised*).

$V_2$  = volume in ml of standard (0.5 N) sodium hydroxide solution used to neutralize the excess of acid in the test with the material,  
 $M$  = mass in g of the *prepared sample* taken for the test, and  
 $M_1$  = percent moisture content determined in A-3.

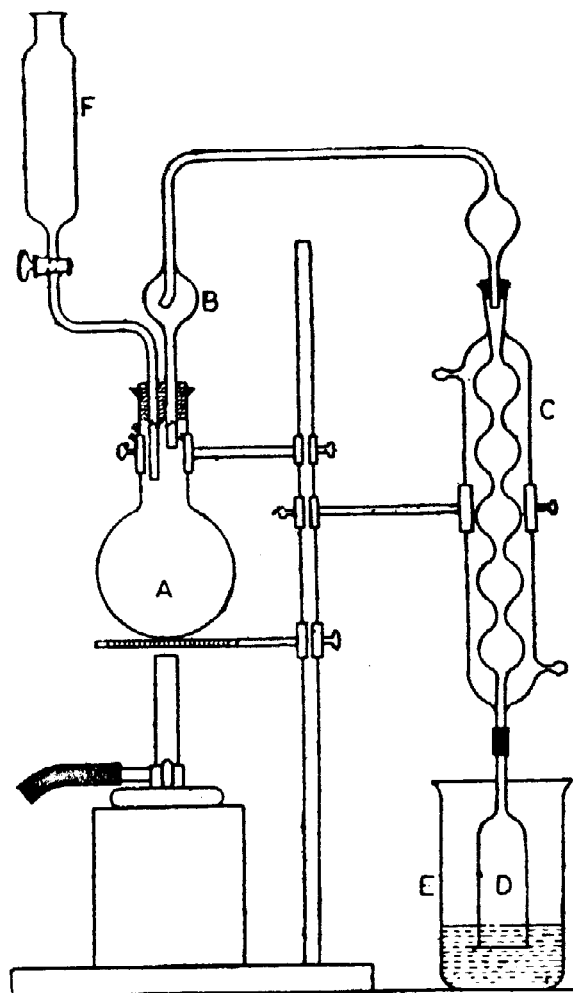


FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF NITROGEN

## A-5. DETERMINATION OF BIURET

**A-5.0 General** — The colour developed by the material with Rochelle salt and copper sulphate is compared with the colour produced by standard biuret solution.

### A-5.1 Apparatus

**A-5.1.1 Spectrophotometer or Photoelectric Colorimeter** — with 4 cm cells.

### A-5.2 Reagents

**A-5.2.1 Alkaline Rochelle Salt Solution** — Dissolve 50.8 g of sodium potassium tartrate tetrahydrate in water containing 40 g of carbonate-free sodium hydroxide. Dilute the solution to one litre and age for one day before use.

**A-5.2.2 Copper Sulphate Solution** — Dissolve 15 g of copper sulphate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in one litre of water and age for one day before use.

**A-5.2.3 Standard Biuret Solution** — Weigh 1.0 g of pure biuret, previously dried at  $105^\circ\text{C}$ ; dissolve this in water and adjust the pH to 7.0 by addition of 0.1 N sulphuric acid solution or 0.1 N sodium hydroxide solution using an indicator paper. Dilute to 500 ml with water. One millilitre of this solution contains 2 mg of biuret.

**NOTE** — Biuret may be purified by washing with 2 N ammonia solution followed by washing with water and acetone and finally drying at  $105^\circ\text{C}$ .

**A-5.3 Procedure** — Pulverize the *prepared sample* (see A-2.1) and dissolve an accurately weighed quantity containing about 20 to 150 mg of biuret, in 25 ml of water. Neutralize the solution to pH 7.0, add 0.2 ml of 2 percent aluminium sulphate solution and 0.2 ml of 0.2 N sodium hydroxide solution. Transfer to a 100 ml volumetric flask and dilute to the mark. Filter the solution through dry filter paper. Transfer a 50 ml aliquot to a 100 ml volumetric flask, add successively from pipettes 20 ml of alkaline Rochelle salt solution and 20 ml of copper sulphate solution with constant swirling. Dilute to the mark, shake vigorously and suspend in a water-bath maintained at  $30 \pm 1^\circ\text{C}$  for 15 to 20 minutes. Shake the flask occasionally while in the water-bath. Remove the solution from the bath and determine its colour scale reading with the spectrophotometer at a wavelength of about 550 nm. Simultaneously, transfer from a burette varying amounts of standard biuret solution to 100 ml volumetric flasks. Adjust the total volume to 50 ml with water and proceed as with the *prepared sample*. Take spectrophotometric readings of the sample solution and of the blank solution at a wavelength of 550 nm using water in the reference cell. Plot a calibration curve of the standard biuret solutions.

**A-5.4 Calculation**

$$\text{Biuret content, percent by mass} = \frac{100 (M_1 - M_2) D}{M}$$

where

$M_1$  = mass in g of biuret found in the aliquot portion of the sample solution,

$M_2$  = mass in g of biuret found in the corresponding aliquot portion of the blank solution,

$D$  = ratio of the volume of the sample solution to that of the aliquot portion taken for colour development, and

$M$  = mass in g of the material taken for the test in **A-5.2**.

**A-6. DETERMINATION OF FREE AMMONIA**

**A-6.0 General** — The material is dissolved in water and titrated with standard acid.

**A-6.1 Reagents**

**A-6.1.1 Standard Hydrochloric Acid** — 0.02 N.

**A-6.1.2 Methyl Red Indicator** — Dissolve one gram of methyl red in 200 ml of rectified spirit (conforming to IS: 323-1955\*).

**A-6.2 Procedure** — Dissolve about 10 g of the *prepared sample* (see **A-2.1**), accurately weighed, in about 50 ml of cold, neutral water. Make up to volume of about 100 ml. Titrate with standard hydrochloric acid using two or three drops of methyl red indicator.

**A-6.3 Calculation**

$$\text{Free ammonia (as NH}_3\text{), percent by mass} = \frac{1.703 V N}{M}$$

where

$V$  = volume in ml of standard hydrochloric acid used in the titration,

$N$  = normality of standard hydrochloric acid, and

$M$  = mass in g of the *prepared sample* taken for the test.

**A-7. DETERMINATION OF AMMONIUM SALTS****A-7.1 Reagents**

**A-7.1.1 Standard Sodium Hydroxide Solution** — 0.1 N.

\*Specification for rectified spirit (*revised*).

**A-7.1.2 Formaldehyde Solution** — 37 percent.

**A-7.1.3 Phenolphthalein Indicator** — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (conforming to IS : 323-1959\*) and dilute with water to 100 ml.

**A-7.2 Procedure** — Weigh accurately  $50.0 \pm 2.0$  g of the *prepared sample* into a 500 ml beaker and dissolve in approximately 250 ml of water. Add a few drops of phenolphthalein solution. If no pink colour develops add 0.1 N sodium hydroxide dropwise until the solution becomes faintly pink. Dilute 10 ml of formaldehyde solution with 90 ml of distilled water in a measuring cylinder and add a few drops of phenolphthalein. Add 0.1 N sodium hydroxide solution dropwise until the formaldehyde solution is faintly pink. Pour the formaldehyde solution into the sample solution and stir to mix. Titrate the mixture with standard sodium hydroxide solution to a faint pink colour.

**A-7.3 Calculation**

$$\text{Ammonium salts (as NH}_3\text{), percent by mass} = \frac{1.703 \times V N}{M}$$

where

$V$  = volume in ml of standard sodium hydroxide used in the titration,

$N$  = normality of standard sodium hydroxide, and

$M$  = mass in g of the *prepared sample* taken for the test.

**A-8. DETERMINATION OF RESIDUE ON IGNITION**

**A-8.1 Procedure** — Ignite a flat-bottomed platinum or silica dish, about 50 mm diameter and 25 mm high, at  $800 \pm 25^\circ\text{C}$ , cool in a desiccator and weigh to the nearest 0.1 mg. Also weigh separately about 100 g of the *prepared sample* to the nearest 0.1 g. Heat the dish over a small flame. Place in it a little of the sample and when it has melted, add the remainder of the sample in small portions, waiting after each addition until all the sample has melted and partially decomposed. Transfer the dish containing the partially decomposed material to an electric furnace controlled at about  $300^\circ\text{C}$ , and raise the temperature slowly to  $800 \pm 25^\circ\text{C}$  at such a rate as to avoid loss by spluttering (about 1 hour). Continue heating until the residue is completely ignited (about 30 minutes). Remove the dish from the furnace, cool in a desiccator and weigh. Repeat the ignition to constant mass. Preserve the dish along with residue for the determination of iron as in A-9.

\*Specification for rectified spirit (*revised*).

**A-8.2 Calculation**

$$\text{Residue on ignition, percent by mass} = 100 \frac{m}{M}$$

where

$m$  = mass in g of the residue, and

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

**A-9. DETERMINATION OF IRON**

**A-9.0 General**—The iron present is reduced to the ferrous state and determined spectrophotometrically or visually using 2, 2'-bipyridyl.

**A-9.1 Apparatus**

**A-9.1.1 Spectrophotometer**—with 1 cm cells. Alternatively Nessler cylinders may be used.

**A-9.2 Reagents**

**A-9.2.1 Dilute Hydrochloric Acid**—approximately 1 N.

**A-9.2.2 Standard Iron Solution**—Dissolve 7.022 g of ammonium ferrous sulphate hexahydrate in a mixture of 600 ml of water and 350 ml of concentrated sulphuric acid and dilute to 1 000 ml with water. Immediately before use dilute 10 ml of this solution to 1 000 ml with water. One millilitre of the diluted solution contains 10  $\mu$ g of iron.

**A-9.2.3 Hydroxylammonium Chloride Solution**—Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

**A-9.2.4 Ammonium Acetate Solution**—Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

**A-9.2.5 2, 2'-Bipyridyl Solution**—Dissolve 1 g of the reagent in 50 ml of water containing 10 ml of dilute hydrochloric acid (1 N) and dilute with water to 100 ml.

**A-9.2.6 Potassium Hydrogen Sulphate**—anhydrous.

**A-9.3 Procedure**

**A-9.3.1 Preparation of Colour Standards**—Into each of a series of eleven 100-ml one-mark volumetric flasks, place respectively such amounts of the standard iron solution as containing from 0 to 500 mg of iron increasing successively by 50  $\mu$ g. Add to each volumetric flask an amount of water sufficient to bring the volume to approximately 50 ml, then add 2 ml of dilute hydrochloric acid, 2 ml of hydroxylammonium chloride solution and 1 ml of 2, 2'-bipyridyl solution. Dilute the contents of each flask to the mark, thoroughly mix and allow to stand for 10 minutes.



Measure the optical density of each solution, at the wavelength of maximum absorption ( approximately 520 nm ) using the solution containing 0 ml of standard iron solution as reference and prepare a calibration chart. Alternatively these standards are used directly for visual comparison.

**A-9.3.2 Determination** — To the residue from the determination of residue on ignition (**A-8**) add 1 g of potassium hydrogen sulphate. Heat the crucible until the contents are completely melted, maintain in a molten state for 10 minutes, and then allow to cool. Add a little water and 5 ml of dilute hydrochloric acid, heat gently until the contents of the crucible have completely dissolved, transfer to a 50 ml one-mark volumetric flask ( filtering if necessary ) and dilute with water to the mark. Pipette into a 100 ml one-mark volumetric flask an aliquot portion of the solution containing between 50 and 500  $\mu\text{g}$  of iron, dilute to about 50 ml, and successively add, mixing after each addition, 2 ml of dilute hydrochloric acid, 2 ml of hydroxylammonium chloride solution and allow to stand for 5 minutes. Add 5 ml of the ammonium acetate solution, mix and add 1 ml of 2, 2' - bipyridyl solution. Dilute to 100 ml with water, mix and allow to stand for 10 minutes.

Measure the optical density of the solution, and of the blank solution at the wavelength used for the calibration using water in the reference cell and read the amount of iron present in the aliquot from the calibration chart.

Alternatively compare the colour of the solution with the series of prepared colour standards in matched Nessler cylinders, noting the iron content of the standard that most nearly matches the test solution.

#### A-9.4 Calculation

$$\text{Iron ( as Fe ), percent by mass} = \frac{100 ( M_1 - M_2 ) D}{M}$$

where

$M_1$  = mass in g of iron found in the aliquot portion of the sample solution,

$M_2$  = mass in g of iron found in the corresponding aliquot of the blank solution,

$D$  = ratio of the volume of the sample solution to that of the aliquot portion taken for colour development, and

$M$  = mass in g of the material taken for the test in **A-8.1**.

## A-10. DETERMINATION OF pH VALUE, ALKALINITY VALUE AND BUFFER VALUE

### A-10.0 Definition

**A-10.0.1 Alkalinity Value** — is the number of millilitres of 0.05 N acid required to lower the pH of a solution of 50 g of urea in 450 ml of water to 8.0.

**A-10.0.2 Buffer Value** — is the number of millilitres of 0.05 N acid required to lower the pH of a solution of urea (see A-10.0.1) from 8.0 to 6.0.

### A-10.1 Apparatus

**A-10.1.1 pH Meter** — with glass and calomel electrode assembly.

**A-10.2 Reagents** — Water conforming to IS:1070-1960\* and with pH value between 6.5 and 6.9 at 25°C shall be used. It shall be freshly boiled and cooled and stored in a container protected from atmospheric contamination.

**A-10.2.1 Buffer Solutions** — in the pH range 6 to 10 (see IS:3225-1965†).

**A-10.2.2 Standard Hydrochloric Acid** — 0.05 N.

### A-10.3 Procedure

**A-10.3.1 pH Value** — Calibrate the pH meter against two buffer solutions covering the range 6 to 10. Weigh about 50 g of the sample to the nearest 0.1 g, transfer to a tall-form 600-ml beaker and dissolve in 450 ml of water, which was previously adjusted to pH 8.0. Mix thoroughly. Adjust the temperature to  $25.0 \pm 0.5^\circ\text{C}$  and determine the pH value.

**A-10.3.2 Alkalinity Value** — If the initial pH of the solution is greater than 8.0, titrate the solution with standard hydrochloric acid, stirring the solution continuously, using a mechanical or magnetic stirrer, adding 0.25 ml portions of the titrant and taking pH readings 10 to 15 seconds after adding it. Continue titration until pH of the solution comes down to 8.0. Construct a graph by plotting pH values against quantity of 0.05 N acid added, in millilitres. From the graph record the alkalinity as the number of millilitres of 0.05 N hydrochloric acid required to lower the initial pH value to 8.0. Use the solution for test in A-10.3.3. If the pH of the urea solution is itself below 8.0, record alkalinity value as nil and use the solution directly for test in A-10.3.3.

\*Specification for water, distilled quality (revised).

†Methods for preparation of buffer solutions.

**A-10.3.3 Buffer Value** — Continue titrating the solution obtained in **A-10.3.2** with standard hydrochloric acid, in exactly the same manner, till the pH comes down to 6.0 and plot the pH values against millilitres of the acid used in the same graph as given in **A-10.3.2**. Record the buffer value as the number of millilitres of 0.05 N hydrochloric acid required to change the pH value from 8.0 to 6.0.

## **A-11. DETERMINATION OF MELTING POINT**

**A-11.1 Procedure** — Determine the melting point by the method prescribed in IS : 5762-1970\*.

# **A P P E N D I X B**

( Clause 4.1 )

## **SAMPLING OF UREA, TECHNICAL**

### **B-1. GENERAL REQUIREMENTS OF SAMPLING**

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.2** The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action. The containers shall be of such size that they are almost completely filled by the sample.

**B-1.3** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

**B-1.4** Sample shall be stored in a cool and dry place.

### **B-2. SCALE OF SAMPLING**

**B-2.1 Lot** — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute a separate lot. In the case of a consignment drawn from a continuous process, 2 000 containers ( or 100 metric tonnes of the material ) shall constitute a lot.

\*Method for determination of melting point and melting range.

**B-2.2** The number of containers to be chosen from a lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

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**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING**

Lot Size	NUMBER OF CONTAINERS TO BE SELECTED
<i>N</i>	<i>n</i>
(1)	(2)
Up to 100	5
101 ,, 300	6
301 ,, 500	7
501 ,, 800	8
801 ,, 1 300	9
1 301 and above	10

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**B-2.3** These containers shall be chosen at random from the lot, and in order to ensure randomness of selection, random sampling procedures given in IS : 4905-1968\* may be followed.

### **B-3. TEST SAMPLES AND REFEREE SAMPLE**

**B-3.1** Draw with an appropriate sampling instrument small portions of the material from different parts of the containers selected, the total quantity taken out from each container being sufficient to conduct the test for all characteristics given in 2.

**B-3.2** Mix thoroughly all portions of the material drawn from the same container to form an individual test sample. Equal quantities from all individual test samples so formed shall be mixed together to form a composite test sample.

**B-3.3** All the individual test samples and the composite test sample shall be divided into three equal parts, thus forming three sets of test samples. These parts shall be immediately transferred to thoroughly dried bottles which shall then be sealed air-tight with glass stopper. These shall be labelled with all the particulars of sampling. One of these sets of test samples shall be sent to the purchaser and another to the supplier.

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\*Methods for random sampling.

**B-3.4 Referee Sample** — The third set of test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee sample and shall be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

#### **B-4. NUMBER OF TESTS**

**B-4.1** Tests for the determination of nitrogen shall be conducted on each of the individual test samples.

**B-4.2** Tests for the remaining characteristics given in 2 shall be conducted on the composite test sample.

#### **B-5. CRITERIA FOR CONFORMITY**

**B-5.1** For nitrogen content, the mean and range of test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

**Range (R)** = The difference between the maximum and the minimum values of the test results.

The lot shall be declared to have satisfied the requirement for total nitrogen if the value of the expression  $(\bar{X} - 0.6 R)$  is equal to or greater than 46.0.

**B-5.2** For the remaining characteristics, the test results on the composite test sample shall satisfy the requirements specified in 2.

**B-5.3** A lot shall be declared as conforming to the specification only when it has satisfied each of the requirements specified in 2.

( Continued from page 2 )

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