भारतीय मानक

Indian Standard

IS 1781 : 2022

यूरिया, तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

Urea, Technical — Specification

(Second Revision)

ICS 71.080.30

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December 2022 **Price Group 9**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian (Second Revision) Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, coal and Related Products Division Council.

This standard was originally published in 1961 and subsequently revised in 1975. In 1961 version, the standard covered the industrial and the fertilizer grades. Later, it was decided to prepare a separate standard for fertilizer grade urea, published as IS 5406.

In first revision covering technical grade urea, the biuret content was lowered since it was observed that manufacturers of technical grade urea were able to comply with the reduced limit and as the consumers wanted as low a limit as possible. Additional requirements were prescribed for ammonium salts, residue on ignition, melting point, alkalinity value and buffer value. The revision was prepared taking into consideration the needs of the plastics and adhesives industries.

The International Organization for Standardization has brought out the following standards on urea for industrial use and assistance has been taken from 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 *p*H 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.

In this (second) revision, alternate method is incorporated for determination of Iron using 1, 10-phenanthroline. A clause is incorporated in sampling process (Annex J), informing the procedure to collect samples from continuous production facilities.

The composition of the Committee, responsible for the formulation of this standard is listed in Annex K.

For the determination of particle size, IS Sieves [*see* IS 460 (Part 1)] have been prescribed, where IS Sieves are not available, equivalent test sieves as judged from aperture size may be used.

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.

Indian Standard

UREA, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

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

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the 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 editions of the standards indicated below:

IS No.	*Title*
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 265 : 2021	Hydrochloric Acid — Specification *(fifth revision)*
IS 460 (Part 1) : 2020	Test Sieves — Specification: Part 1 Wire Cloth Test Sieves(*fourth revision*)
IS 915 : 2012	Laboratory glassware — One-mark volumetric flasks *(third revision)*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
IS 5762 : 1970	Methods for determination of melting point and melting range.
IS 15557 : 2005	Methods for preparation of buffer solutions and methods for determination of *p*H

3 REQUIREMENTS

3.1 Description

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

3.2 In the form of prills, the material shall pass through 4.00 mm IS sieve and not less than 90 percent by mass shall be retained on 0.85 mm IS Sieve [*see* IS 460 (Part 1)].

3.3 The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in col 4 and col 5 of the Table 1.

3.4 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 Annex H:

a) Alkalinity value, 2.0 *Max*; and

b) Buffer value, 4.0 *Max.*

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in polyethylene lined jute or HDPE or PP bags or in such other containers as agreed to between the purchaser and the supplier.

Table 1 Requirements for Urea, Technical

(*Clause* 3.3 *and* 6.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by mass, *Max*	0.5	A	—
ii)	Nitrogen, percent by mass (on dry basis), *Min*	46.0	B	—
iii)	Biuret, percent by mass, *Max*	0.8	C	—
iv)	Free ammonia (*as* NH_3), percent by mass, *Max*	0.015	D	—
v)	Ammonium salts (*as* NH_3), percent by mass, *Max*	0.01	E	—
vi)	Residue on ignition, percent by mass, *Max*	0.002	F	—
vii)	Iron (*as* Fe), percent by mass, *Max*	0.000 2	G	—
viii)	*p*H of 10 percent solution	7.0 to 9.5	H	—
ix)	Melting point, ºC	131 to 133	—	IS 5762

4.2 Marking

4.2.1 The containers shall be securely closed and marked with the following information:

a) Name of the material;

b) Name of manufacturer and his recognized trademark, if any;

c) Year of manufacture;

d) Mass (in kg) of the material in the container; and

e) Any other statutory requirements.

4.2.2 *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.

5 SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex J.

6 TEST METHODS

6.1 Preparation of sample

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.

6.2 Test shall be conducted as prescribed in col **4** and col **5** of Table 1.

6.3 Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS1070), shall be employed in tests.

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

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MOISTURE

A-1 PROCEDURE

Carry out the determination as prescribed in IS 2362, using a weighed quantity of the prepared sample (**6.1**) containing 10 mg to 50 mg of water.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF NITROGEN BY KJELDAHL METHOD

B-1 APPARATUS

The apparatus, as assembled, is shown in Fig. 1. It consists of a round bottom kjeldahl flask *A* of 500 ml or suitable 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.

B-2 REAGENTS

B-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.

B-2.2 Concentrated Sulphuric Acid (*see* IS 266).

B-2.3 Standard Sulphuric Acid, 0.5 N.

B-2.4 Methyl Red-Methylene Blue Mixed Indicator

Dissolve 0.1 g of methyl red in 50 ml of rectified spirit (*see* IS 323) and add 0.05 g of methylene blue. When it has dissolved, dilute to 100 ml with the same rectified spirit.

B-2.5 Sodium Hydroxide Solution, approximately 45 percent (*m*/*v*).

B-2.6 Standard Sodium Hydroxide Solution, 0.5 N.

B-3 PROCEDURE

B-3.1 Weigh accurately about 0.5 g of the prepared sample (**6.1**) 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*. Clash 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.

B-3.2 Carry out a blank using all reagents in the same quantities but without the material to be tested.

B-4 CALCULATION

Nitrogen, percent by mass (on dry basis)

$$= \frac{70.04\,(V_1 - V_2)}{M(100 - M_1)}$$

where

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

V_2 = volume of standard (0.5 N) sodium hydroxide solution used to neutralize the excess of acid in the test with the material, ml;

M = mass of the prepared sample taken for the test in **B-3**, g; and

M_1 = percent moisture content determined in Annex A**.**

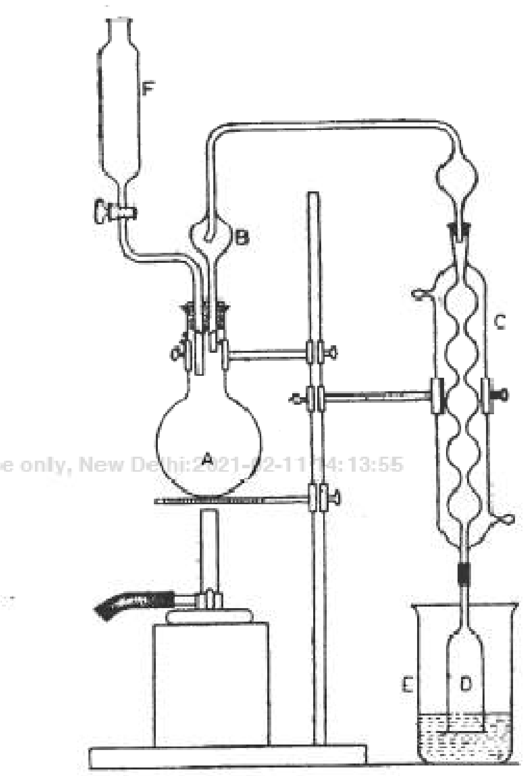

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF NITROGEN

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF BIURET

C-1 GENERAL

The colour developed by the material with Rochelle salt and copper sulphate is compared with the colour produced by standard biuret solution.

C-2 APPARATUS

C-2.1 Spectrophotometer or Photoelectric Colorimeter, with 40 mm to 100 mm cell.

C-3 REAGENTS

C-3.1 Alkaline Rochelle Salt Solution

Dissolve 50.8 g of sodium potassium tartrate tetra hydrate in water containing 40 g of carbonate-free sodium hydroxide. Dilute the solution to 1 l and age for one day before use.

C-3.2 Copper Sulphate Solution

Dissolve 15 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in 1 l of water and age for one day before use.

C-3.3 Standard biuret Solution

Weigh 1.0 g of pure biuret, previously dried at 105 °C; dissolve this in water and adjust the *p*H 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. 1 ml 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 ºC.

C-4 PROCEDURE

Pulverize the prepared sample (**6.1**) and dissolve an accurately weighed quantity containing about 20 mg to 150 mg of biuret, in 25 ml of water. Neutralize the solution to *p*H 7.0, add 0.2 ml of 2 percent aluminum 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 °C ± 1°C for 15 min to 20 min. 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.

C-5 CALCULATION

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

where

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

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

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

M = mass of the material taken for the test in **C-4**, g.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF FREE AMMONIA

D-1 GENERAL

The material is dissolved in water and titrated with standard acid.

D-2 REAGENTS

D-2.1 Standard Hydrochloric Acid, 0.02 N.

D-2.2 Methyl Red Indicator

Dissolve one gram of methyl red in 200 ml of rectified spirit (*see* IS 323).

D-3 PROCEDURE

Dissolve about 10 g of the prepared sample (**6.1**), 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.

D-4 CALCULATION

Free ammonia $(as\ \mathrm{NH3})$, percent by mass

$$= \frac{1.703\ V\mathrm{x}N}{M}$$

where

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

N = normality of standard hydrochloric acid; and

M = mass of the prepared sample taken for the test in **D-3**, g.

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF AMMONIUM SALTS

E-1 REAGENTS

E-1.1 Standard Sodium Hydroxide Solution, 0.1 N.

E-1.2 Formaldehyde Solution, 37 percent.

E-1.3 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323) and dilute with water to 100 ml.

E-2 PROCEDURE

Weigh accurately 50.0 g ± 2.0 g of the prepared sample (**6.1**) 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.

E-3 CALCULATION

$$\text{Ammonium salts } (as\ \text{NH3}), \text{ percent by mass} = \frac{1.703\ \times V \text{ x } N}{M}$$

where

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

N = normality of standard sodium hydroxide; and

M = mass of the prepared sample taken for the test in **E-2**, g.

ANNEX F

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF RESIDUE ON IGNITION

F-1 PROCEDURE

Ignite a flat-bottomed platinum or silica dish, about 50 mm diameter and 25 mm high, at 800 °C ± 25 ºC, cool in a desiccators 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 ºC and raise the temperature slowly to 800 °C ± 25°C at such a rate as to avoid loss by spluttering (about 1 h). Continue heating until the residue is completely ignited (about 30 min).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 Annex G.

F-2 CALCULATION

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

where

m = mass of the residue, g; and

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

ANNEX G

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF IRON

G-1 METHOD USING 2,2’-BIPYRIDYL

G-1.1 General

The iron present is reduced to the ferrous state and determined spectrophotometrically or visually using 2, 2’-bipyridyl.

G-1.2 Apparatus

G-1.2.1 *Spectrophotometer*, with 1 cm cells. Alternatively, nessler cylinders may be used.

G-1.3 Reagents

G-1.3.1 *Dilute hydrochloric acid*, 1 N.

G-1.3.2 *Standard iron solution*

Dissolve 7.022 g of ammonium ferroussulphate 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. 1 ml of the diluted solution contains 10 µg of iron.

G-1.3.3 *Hydroxylammonium chloride solution*

Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

G-1.3.4 *Ammoniumacetate solution*

Dissolve 30 g of ammonium acetatein water and dilute to 100 ml.

G-1.3.5 *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.

G-1.3.6 *Potassium hydrogen sulphate*, anhydrous.

G-1.4 Procedure

G-1.4.1 *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 mg to 500 mg of iron increasing successively by 50 µg. 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 min.

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.

G-1.4.2 *Determination*

To the residue from the determination of residue on ignition (Annex F) add 1 g of potassium hydrogen sulphate. Heat the crucible until the contents are completely melted, maintain in a molten state for 10 min, 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 µg and 500 µg 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 min. 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 min.

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.

G-1.4.3 *Calculation*

$$\text{Iron } (as \text{ Fe}), \text{percent by mass} = \frac{100(\mathrm{M}_1 - \mathrm{M}_2)\mathrm{D}}{\mathrm{M}}$$

where

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

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

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

M = mass of the material taken for the test in **F-1,** g.

G-2 METHOD USING 1, 10-PHENANTHROLINE

G-2.1 General

Iron (Fe^{+2}) reacts with 1,10-phenanthroline to form an orange-red complex $[(C_{12}H_8N_2)_3Fe]^{2+}$. The colour intensity is independent of the acidity in the *p*H range 2-9 and is stable for long periods. The complex follows Beers' Law closely and its absorbance is very stable for long period.

Ferric iron is reduced with hydroquinone and *p*H is adjusted with an acetate buffer. The solution, slightly acidic with Hydrochloric acid, is treated with 1,l0-phenanthroline, and buffered with sodium citrate at a *p*H of 4. The iron - phenanthroline complex is measured at 510 nm against reagent blank.

G-2.2 Apparatus

G-2.2.1 *One-mark volumetric flasks*, 1 000 ml capacity (*see* IS 915).

G-2.2.2 *One-mark volumetric flasks*, 500 ml capacity (*see* IS 915).

G-2.2.3 *One-mark volumetric flasks*, 50 ml capacity (*see* IS 915).

G-2.2.4 *UV-Vis spectrophotometer*

G-2.2.5 *Glass cell*, of 50 mm path length, 2 Nos.

G-2.2.6 *Standard micro burette,* 10 ml capacity.

G-2.3 Reagents

G-2.3.1 *Hydrochloric Acid*, relative density 1.16 (*see* IS 265).

1:1 HCL, dilute hydrochloric acid with equal volume of demineralized water.

G-2.3.2 *Hydroquinone solution*, 1 percent hydroquinone solution in demineralized water. Kept in brown bottle and discard when turbid.

G-2.3.3 *Sodium citrate solution*, 25 percent of Sodium citrate solution in demineralized water.

G-2.3.4 *1, 10-Phenanthroline*, 0.10 percent of 1, 10-phenanthroline in demineralized water.

G-2.3.5 *Standard iron solution*

Dissolve 3.511 gm of ammonium ferrous sulphate [$(NH_4)_2Fe(SO_4)_2.6H_2O$] in 500 ml demineralized water containing 10 ml concentrated sulfuric acid (H_2SO_4).

1 ml = 1 000 µg Iron ——— Solution (A)

Take 2 ml of solution (A) with micro burette in 1 000 ml volumetric flask. Make it up with demineralized water.

It will give 1 ml = 2 µg —— Solution (B)

G-2.4 Procedure

To the basin containing the residue from the determination of residue on ignition (**F-1**), add 5 ml of concentrated hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a glass rod until all the residue has dissolved. Allow to cool, transfer to the 50 ml volumetric flask, (if high iron content is expected, then make up the volume to 50ml and take suitable aliquot into another 50ml volumetric flask). Add 1 ml 1:1 HCl, 1.0 ml of 1 percent hydroquinone solution, stir well and wait for 5 min. Add 5 ml of 0.1 percent 1, 10-phenanthroline and 5 ml of 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with demineralized water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength.

G-2.5 Calculation

$$\text{Iron as Fe, ppm} = \frac{[\mu\text{g of Fe obtained from spectrophotometer x dilution factor (if applicable)}]}{M \text{ x } aliquot\ of\ sample\ stock}$$

where

M = mass in g of sample taken for the determination of residue on evaporation (**F-1**)

Iron (*as* Fe) percent by mass = Iron (*as* Fe), ppm / 10 000

G-2.6 Calibration of Spectrophotometer for Iron

G-2.6.1 *Calibration range,* 0 to 10 µg.

G-2.6.2 *Procedure*

Take 1,2,3, 4 and 5 ml of standard solution B (**G-2.3.5**) in 50 ml volumetric flask keeping sixth as blank. Add 1 ml of 1:1 HCl, add 1.0 ml of 1 percent hydroquinone solution, stir well and wait for 5 min. Add 5 ml of 0.1 percent 1,10-phenanthroline and 5 ml of 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with demineralized water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength. Measured absorbance data and slope value are mentioned.

NOTES

1 Other type of spectrophotometric method may be used, provided slope determination for the specified range is established and accuracy level as specified in this standard is achievable.

2 For analysis of iron instrumental method of analysis like AAS, ICP may be used, provided calibration of instrument is done in accordance with detection levels as necessary with requirements of this standard.

ANNEX H

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF *p*H VALUE, ALKALINITY VALUEAND BUFFER VALUE

H-1 TERMINOLOGY

H-1.1 Alkalinity Value — Is the number of ml of 0.05 N acid required to lower the *p*H of a solution of 50 g of urea in 450 ml of water to 8.0.

H-1.2 Buffer Value — Is the number of ml of 0.05 N acid required to lower the *p*H of a solution of urea (**H-1.1**) from 8.0 to 6.0.

H-2 APPARATUS

H-2.1 *p*H Meter, With glass and calomel electrode assembly.

H-3 REAGENTS

Water conforming to IS 1070 and with *p*H value between 6.5 and 6.9 at 25°C shall he used. It shall be freshly boiled and cooled and stored in a container protected from atmospheric contamination.

H-3.1 Buffer Solutions, in the *p*H range 6 to 10 (*see* IS 15557).

H-3.2 Standard Hydrochloric Acid*,* 0.05 N.

H-4 PROCEDURE

H-4.1 *p*H Value

Calibrate the *p*H 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 *p*H 8.0. Mix thoroughly. Adjust the temperature to 25.0 °C ± 0.5 °C and determine the *p*H value.

H-4.2 Alkalinity Value

If the initial *p*H 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 *p*H readings 10 to 15 sec after adding it. Continue titration until *p*H of the solution comes down to 8.0. Construct a graph by plotting *p*H values against quantity of 0.03 N acid added, in ml. From the graph record the alkalinity as the number of ml of 0.05 N hydrochloric acid required to lower, the initial *p*H value to 8.0. Use the solution for test in **H-4.3**. If the *p*H of the urea solution is itself below 8.0, record alkalinity value as nil and use the solution directly for test in **H-4.3**.

H-4.3 Buffer Value

Continue titrating the solution obtained in **H-4.2** with standard hydrochloric acid, in exactly the same manner, till the *p*H comes down to 6.0 and plot the *p*H values against ml of the acid used in the same graph as given in **H-4.2**. Record the buffer value as the number of ml of 0.05 N hydrochloric acid required to change the *p*H value from 8.0 to 6.0.

ANNEX J

(*Clause* 5.1)

SAMPLING OF UREA, TECHNICAL

J-1 GENERAL REQUIREMENTS OF SAMPLING

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

J-l.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

J-l.3 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.

J-l.4 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.

J-1.5 Sample shall be stored in a cool and dry place.

J-2 SCALE OF SAMPLING

J-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 tons of the material) shall constitute a lot.

J-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 co1 2 and 3 of Table 2.

Table 2 Number of containers to be selected for sampling

(*Clause* J-2.2)

Sl No. (1)	**Lot Size** *N* (2)	**Number or containers to be selected** *N* (3)
i)	up to 100	5
ii)	101 to 300	6
iii)	301 to 500	7
iv)	501 to 800	8
v)	801 to 1 300	9
vi)	1 301 and above	10

J-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 may be followed.

J-2.4 For continuous production facilities, samples shall be collected from conveyor belt periodically and combined in equal proportion so as to constitute sample representing a lot of respective production capacity.

J-3 TEST SAMPLES AND REFEREE SAMPLE

J-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 **3**.

J-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.

J-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.

J-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.

J-4 NUMBER OF TESTS

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

J-4.2 Tests for the remaining characteristics given in **3** shall be conducted on the composite test sample.

J-5 CRITERIA FOR CONFORMITY

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

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

where

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 ($\overline{X}$ - 0.6 R) is equal to or greater than 46.0.

J-5.2 For the remaining characteristics, the test results on the composite test sample shall satisfy the requirements specified in **3**.

J-5.3 A lot shall be declared as conforming to the specification only when it has satisfied each of the requirements specified in **3**.

ANNEX K

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	DR C. V. RODE **(*Chairperson*)**
Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (*Alternate*)
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL (*Alternate*)
CSIR- Central Drug Research Institute (CDRI), Lucknow	DR SANJEEV KANOJIYA
Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI MAHINDER SINGH
Deepak Fertilize, New Delhi	DR L. B. YADAWA SHRI SURESH AMLE (*Alternate*)
Deepak Phenolics Limited, Vadodara	SHRI DHARMESH SIDDHAPURIA SHRI SANDIP KUMAR PANDYA (*Alternate*)
Dow Chemical International Private Limited, Mumbai	SHRI V. MOHANDOSS SHRI GOVIND GUPTA (*Alternate*)
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR SHRI APPASAHEB J. WANI (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Gujarat	SHRI R. M. PATEL SHRI C. S. PATEL (*Alternate*)

Organization	*Representative(s)*
Hindustan Organic Chemicals Limited (HOCL), Mumbai	SHRI DELEEP KUMAR K. SHRI V. MOHAN (*Alternate*)
India Glycols Limited, Uttarakhand	SHRI S. R. SONI SHRI ALOK SINGHAL (*Alternate*)
Indian Chemical Council (ICC), Mumbai	DR MRITUNJAY CHAUBEY SHRI J SEVAK (*Alternate*)
Jubilant Life Sciences Limited, Uttar Pradesh	SHRI HARI MOHAN LOHANI
Laxmi Organic Industries, Mumbai	SHRI J. P. SURYAVANSHI DR VIJAY S. MISHRA (*Alternate*)
Department of Chemicals & Petrochemicals, Ministry of Chemicals & Fertilizers, New Delhi	SHRI O. P. SHARMA SHRI VARUN SINGH POONIA (*Alternate*)
National Chemical Laboratory (NCL), Pune	DR RAVINDAR KONTHAM DR UDAYA KIRAN MARELLI (*Alternate*)
National Test House (NTH) , Ghaziabad	SHRI DEBASHIS SAHA DR GOPAL KRISHAN (*Alternate*)
Reliance India Limited (RIL), Mumbai	SHRI K. K. SREERAMACHANDRAN SHRI VASANT WARKE (*Alternate*)
United Phosphorus Limited (UPL), Mumbai	SHRI M. D. VACHHANI
In Personal Capacity	DR MAYUR J. KAPADIA
Director General, BIS	SHRIMATI NAGAMANI. T, SCIENTIST 'E'/DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS DEPARTMENT) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

MS ADITI CHOUDHARY

Scientist 'B'/ASSISTANT DIRECTOR

(PETROLEUM, COAL AND RELATED PRODUCTS DEPARTMENT) BIS

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This Indian Standard has been developed from Doc No.: PCD 09 (18178).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi