भारतीय मानक
Indian Standard

IS 8058 : 2018

पीरिडीन — विशिष्टि

(पहला पुनरीक्षण)

Pyridine — Specification

(First Revision)

ICS 71.080.30

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

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on recommendation of the Organic Chemicals, Alcohols and Allied Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

Pyridine is used as solvent in dyestuff industry, pharmaceutical industry and as a laboratory reagent. It is also used in the manufacture of drug intermediates like 2-aminopyridine, 2,6-diaminopyridine, pheniramine and zelan which is used in waterproofing fabrics. It is also finds use in making decapryn, ritalin and pipradol, and pesticides. Pyridine base as denaturants is covered in IS 4117.

This standard was first published in 1976.

In this revision following major changes have been made:

a) Requirements of relative density, pyridine content, sulphates and moisture content have been modified and requirement of relative density has been specified for grade 2 and 3. Requirements for color has been added. As alternate to ammonium compounds requirement of free ammonia has been included.

b) Method for test of pyridine content has been updated. Alternate test methods for free ammonia and alternate test methods for chloride, and copper have been included to facilitate accurate and faster testing.

Amendment number 1 to IS 8058 : 1976 dated February 1989 has also been incorporated in this revision of the standard.

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 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

PYRIDINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for pyridine.

2 REFERENCES

The standards given below 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 subject are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
915 : 2012	Laboratory glassware — One-mark volumetric flasks (*third revision)*
1070 : 1992	Reagent grade water (*third revision)*
1260 (Part 1) : 1973	Pictorial marking for handling and labeling of goods: Part 1 Dangerous goods (*first revision)*
2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
4161 : 1967	Nessler cylinders
4905 : 2015	Methods for random sampling *(first revision)*
5298 : 2013	Method for determination of distillation range and of distillation yield (*first revision)*
8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale *(second revision)*

3 GRADES

There shall be three grades of the material, namely:

a) Grade 1 — Suitable for use as a reagent in analytical chemistry and for special pharmaceutical preparations.

b) Grade 2 — Suitable for use in the manufacture of drug intermediates.

c) Grade 3 — Mainly used as a solvent in dyestuff industry.

4 REQUIREMENTS

4.1 Description

The material shall be clear liquid free from foreign matter. It shall also have a characteristic disagreeable odour of pyridine.

4.2 Solubility

The material shall be miscible with water, alcohol, chloroform and ether. It shall also pass the following test:

Mix 10 parts of the material in 90 parts of water and shake the mixture. The material shall be clear and homogeneous.

4.3 Identity and Purity

From chromatographic examination (*see* Annex B), the material shall appear identical or at least equivalent to the standard material.

4.4 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in col 6 of Table 1.

5 PRECAUTIONS IN STORING AND HANDLING

5.1 As pyridine is flammable, necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions taken at all times to prevent accident by fire or explosion. It shall be stored in a cool place. Open flames and smoking shall not be allowed where it is stored or handled. Storage vessels shall be vented to safe atmosphere.

5.2 Except when they are opened for the purpose of cleaning and rendering them free from pyridine vapours, all empty tanks and other containers shall be securely closed.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in mild steel drums. The gaskets for the bungs shall be of high density polythene.

6.2 Marking

6.2.1 Each container shall be suitably marked with the following information:

a) Name and grade of the material;

b) Net mass of the material in the container;

c) Name of the manufacturer and his recognized trade-mark, if any;

d) Batch number or lot number, in code or otherwise; and

e) The symbol given in Fig. 5 of IS 1260 (Part 1), and the words 'HARMFUL, VAPOUR FLAMMABLE. KEEP IN COOL PLACE' in capitals.

6.2.2 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

6.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made there under. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

7 SAMPLING

The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex M.

8 QUALITY OF REAGENTS

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

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

TABLE 1 REQUIREMENTS FOR PYRIDINE

(*Clause* 4.4)

Sl No.	Characteristic	Requirements for			Method of Test, Ref to IS/Annex
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Relative density[1)] at 25^0 C/25^0 C	0.984 to 0.986	0.984 to 0.986	0.984 to 0.986	A
ii)	Pyridine content, percent by mass, M*in*	99.9	99.5	98.5	B
iii)	Boiling range at 760 mm Hg	Minimum 97 percent by volume, shall distill in between 115^0 - 116ºC	Minimum 97 percent by volume, shall distill in between 115^0 - 116ºC	Minimum 97 percent by volume, shall distill in between 115^0 - 116ºC	C
iv)	Residue on evaporation, percent by mass, *Max*	0.002	0.002	—	D
v)	Ammonium compounds (as NH_4), percent by mass, *Max*	0.002	—	—	E
	or				
	Ammonium compounds (as Free Ammonia), percent by mass, *Max*	0.002	—	—	F
vi)	Chlorides (as Cl), percent by mass, *Max*	0.0010	—	—	G
vii)	Copper (as Cu), percent by mass, *Max*	0.0005	—	—	H
viii)	Oxidizable substances	To pass test	To pass test	—	J
ix)	Sulphates (as SO_4), percent by mass, *Max*	0.0005	—	—	K
x)	Moisture content, percent by mass, *Max*	0.10	0.15	0.25	L
xi)	Colour, HU (Pt-Co scale), Max.	15	25	40	IS 8768

[1)] Relative density is the term adopted by ISO for specific gravity with water as reference substance.

ANNEX A

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

DETERMINATION OF RELATIVE DENSITY

A-1 OUTLINE OF THE TEST METHOD

In this method, mass of equal volumes of the material and water are compared at 25°C using a relative density bottle.

A-2 APPARATUS

A-2.1 Relative Density Bottle — 25 ml capacity.

A-2.2 Water Bath — Maintained at 25.0 ± 2°C.

A-2.3 Thermometer — Any convenient thermometer of a suitable range with 0.1 or 0.2°C subdivisions.

A-3 PROCEDURE

A-3.1 Clean and dry the relative density bottle. Weigh and fill with recently boiled and cooled water at 25°C. Fill to over-flowing by holding the relative density bottle on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper, immerse in the water bath maintained at 25 ± 0.2°C and hold for 30 min. Remove the relative density bottle from the bath and clean and dry it thoroughly and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-3.2 Calculation

$$\text{Relative density, at 25°C / 25°C} = \frac{A - B}{C - B}$$

where,

A = mass in g, of the relative density bottle with the material at 25°C;

B = mass in g, of the empty relative density bottle; and

C = weight in g, of the relative density bottle with water at 25°C.

ANNEX B

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

DETERMINATION OF PYRIDINE CONTENT

B-0 OUTLINE OF THE METHOD

The content of pyridine along with other components is determined by gas chromatography.

B-1 GENERAL

The chromatographic conditions given here are for guidance only.

B-2 APPARATUS

B-2.1 Gas Chromatograph — With flame-ionization detector using capillary column

B-2.2 Data Acquisition System — A system capable of acquiring chromatographic data and integrating chromatographic peaks.

B-2.3 Column — CP-WAX-52CB, 60 m long, 0.25 mm ID, film thickness df = 0.25 µm.

B-3 TEST SUBSTANCES

B-3.1 Reference Working Standard of Pyridine

B-3.2 Reference Working Standard of Alpha Picoline

B-3.3 Reference Working Standard of Gamma Picoline

B-3.4 Pentadecane (>99 Percent) for Internal Standard

B-4 PROCEDURE

B-4.1 Operating Parameters of Gas Chromatograph

B-4.1.1 *Injector temp* : 240°C

B-4.1.2 *Detector temp* : 250°C

B-4.1.3 *Carrier flow (N2)* : 1.5 ml/min

B-4.1.4 *Split Ratio* : 1: 100

B-4.1.5 *Make up flow (N2)* : 20 ml/min

B-4.1.6 *Oven temp-1* : 75°C for 18 min

B-4.1.7 *Oven temp-2* : 200°C for 6 min

B-4.1.8 *Programming rate* : 10°C /min

B-4.1.9 *Total run time* : 36.5 min

B-4.1.10 *Range* : 20

B-4.1.11 *Attenuation* : –3

B-4.2 Standard and Test Sample Preparation

B-4.2.1 *For Chromatographic Purity*

Standard and test sample, inject 0.2 µl.

B-4.2.2 *Preparation of Internal Standard (Pentadecane) Solution*

Weigh and transfer accurately about 1000 mg of pentadecane (internal standard) in a 100 ml volumetric flask. Add Methanol to dissolve and dilute the content to the volume. If required sonicate till stock solution becomes clear (concentration 10 mg/ml) and preserve in a cool place. Pipette out 4.0 ml of the above solution into another 100 ml volumetric flask and dilute to the mark with methanol. Final concentration of pentadecane is about 0.4 mg/ml.

B-4.2.3 *Preparation of Standard Solution for Calibration*

Weigh accurately (nearest to ± 0.5 mg) 980 mg of certified/working reference standard of pyridine and 10 mg each working reference sample of alpha picoline, gamma picoline in a clean and dry vial and mix thoroughly. Weigh accurately about 400 mg (nearest to ± 0.5 mg) of the above synthetic mixture into another 10 ml volumetric flask and accurately add 10.0 ml of internal standard by pipette. Dissolve and mix the contents. Adjust the concentration of the sample of peak is off scale.

B-4.2.4 *Test Sample Preparation*

0.2 µl neat of working standard and test sample.

B-4.3 Method

B-4.3.1 Condition the column at 220° C for half an hour. Allow the gas chromatograph to equilibrate at 75°C and obtain a steady baseline before proceeding with analysis. Following is the recommended sequence for analysis and evaluating suitability of the system:

a) Inject 1.0 µl of blank (methanol) — Single injection
b) Inject 1.0 µl of calibration solution — Duplicate injection
c) Inject 0.2 µl (neat) of standard and test sample solution — Single injection

Evaluate system suitability as per Table 2.

Table 2 System Suitability Criteria

Sl No	Method Performance Parameter	Criteria
1.	Diluent blank has no significant interfering peaks. Subtract the blank's peak area from the standard solution for calibration solution.	
2.	Tailing factor for all analyte should	Not more than 2.5
3.	Resolution Between of all impurities	Should not be < 1.5

B-4.4 Calculation

B-4.4.1 *Chromatographic Purity:*

$$\text{Pyridine (percent by area)} = \frac{\text{Peak area of Pyridine obtained in test sample}}{\text{Sum of the area of all the peaks obtained in Test sample}} \times 100$$

B-4.4.2 *Pyridine Content (Percent by Mass)*

B-4.4.2.1 *Calculate the calibration factor or response factor* — By corrected area normalization method

B-4.4.2.2 Calculate the area/mass (A/M) ratio by dividing the area of each peak by its mass as under:

Component	*Mass, Percent(M)*	*Area*	*A/M* Ratio
Pentadecane	1.0	A_1	$A_1/1.0 = K$
Pyridine	97.0	A_2	$A_2/97.0 = L$
Alpha picoline	1.0	A_3	$A_3/1.0 = M$
Gamma picoline	1.0	A_4	$A_4/1.0 = N$

B-4.4.2.3 *Calculate actual mass percent in calibration solution A*

Set arbitrarily internal standard response factor to 1.0 and find response factor of other components as follows:

Component	*Slope*	*Response Factor*
Pentadecane	K/K	1.0
Pyridine	K/L	Put the value obtained
Alpha picoline	K/M	Put the value obtained
Gamma picoline	K/N	Put the value obtained

B-4.4.2.4 Multiply the areas by their response factor to get the true areas of the peaks. Add up the area to get total true area and calculate as per following:

$$\text{Component 'n' in the sample (percent by Mass)} = \frac{An \times (100 - m)}{At}$$

where,

An = True peak area of component 'n',

m = percent of water in the sample, and

At = total true peak area

B-4.4.2.5 *Typical retention time of other components present in pyridine sample*

Sl No.	*Component Name*	*Approx. RT (in minutes)*	*Type of Component*
1	Pyridine	10.5	Finished Product
2	Alpha picoline	11.9	Impurity
3	Gamma picoline	17.4	Impurity
4	Pentadecane	25.1	Internal Standard

ANNEX C

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

DETERMINATION OF BOILING RANGE

C-1 PROCEDURE

C-1.1 Determine the boiling range by the procedure prescribed in IS 5298, applying the following corrections:

C-1.2 Correction of thermometer reading

C-1.2.1 *Error of Scale*

In all thermometer readings, make the corrections as described on the certificate of the instrument.

C-1.3 Correction of Barometric Pressure

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If, however, the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall be corrected as follows:

a) For every 27 mm above 760 mm Hg, subtract 1°C from the specified temperature; and

b) For every 27 mm below 760 mm Hg, add 1ºC to the specified temperature.

Note — These corrections are valid only for pressure above 700 mm Hg.

ANNEX D

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

DETERMINATION OF RESIDUE ON EVAPORATION

D-1 APPARATUS

D-1.1 Porcelain Basin — 150 ml capacity.

D-1.2 Oven — Capable of maintaining temperature of 105 ± 2ºC.

D-2 PROCEDURE

Weigh the porcelain basin. Cool the material to 0ºC and by means of a pipette with rubber bulb aspirator, transfer 100 ml of the material to the tare porcelain basin. Allow the material to evaporate at room temperature and then heat the residue in the oven at 105 ± 2ºC for 30 min. Cool in a desiccator and weigh accurately.

D-3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M1 - M}{V \times d} \times 100$$

where,

M1 = mass in g, of the basin with the residue;

M = mass in g, of the basin;

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

d = relative density of the material as determined under Annex A.

ANNEX E

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

TEST FOR AMMONIUM COMPOUNDS

E-0 OUTLINE OF THE METHOD

The solution of the sample is treated with sodium hydroxide and filtered. To the filtrate, Nessler solution is added and the colour produced is compared with that produced in a control test containing definite amount of ammonium salt.

E- 1 REAGENTS

E -1.1 Sodium Hydroxide Solution — 10 percent (*m/v*)

E-1.2 Nessler Solution

Dissolve 143 g of sodium hydroxide in 700 ml of water. Dissolve 50 g of red mercuric iodide and 40 g of potassium iodide in 200 ml of water. Pour the iodide solution into hydroxide solution and dilute to 1 000 ml. Allow to settle and use the clear supernatant liquid.

E-1.3 Standard Ammonium Chloride Solution

Dissolve 0.296 g of ammonium chloride in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of ammonium (as NH_4).

E-2 PROCEDURE

E-2.1 Weigh 1.000 g of the material and dilute to 45 ml and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible, previously washed with sodium hydroxide solution. Dilute with water to 100 ml and add 2 ml of Nessler solution. In a control test, having an equal volume (100 ml) of the solution and containing 2 ml of standard ammonium chloride and 15 ml of sodium hydroxide solution, add 2 ml of Nessler solution.

E-2.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced with the material is not darker than that produced with the standard solution.

ANNEX F

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

DETERMINATION OF AMMONIUM COMPOUNDS AS FREE AMMONIA

F-1 REAGENTS

F-1.1 Hydrochloric Acid — 0.01 N

F-1.2 Phenolphthalein Indicator — 0.1 percent

F-2 PROCEDURE

F-2.1 Take 2.0 g of sample in 10 ml carbon dioxide free water (boiled and cooled distilled water) in 100 ml flask; add 0.1 ml phenolphthalein indicator and 2 ml (approximately 2.0 g) of pyridine. If pink color appears then titrate it with hydrochloric acid (0.01 N) up to the disappearance of pink color. Note the volume of hydrochloric acid consumed. Do the blank test with 10 ml water and 0.1 ml phenolphthalein indicator

F-2.2 Calculation

Calculate the mass of free ammonia by using the following formula:

$$\text{Free ammonia, percent by mass} = \frac{1.7 \times N \times (V1 - V2)}{M}$$

where,

N = Normality of hydrochloric acid;

V_1 = volume, in ml, of standard hydrochloric acid required for sample;

V_2 = volume, in ml, of standard hydrochloric acid required for blank; and

M = mass of the sample taken for test.

ANNEX G

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

DETERMINATION OF CHLORIDE

G-1 GENERAL

Two methods have been prescribed for the determination of chloride in pyridine. Both the methods can be used for determination of chloride on routine basis. However, in case of dispute, method 1 shall be treated as a referee method.

G-2 METHOD 1

G-2.1 Reagents

G-2.1.1 *Dilute Nitric Acid*

G-2.1.2 *Silver Nitrate Solution* — 5 percent (*m/v*)

G-2.1.3 *Methanol Solution* — Mix methanol (AR grade) and water in the ratio of 70:30.

G-2.1.4 *Standard Chloride Solution* — Dissolve 0.1649 g of sodium chloride in water and make the volume to 100 ml. Pipette out 15 ml of this solution in another 50 ml volumetric flask and dilute with water to the mark. 1 ml of the final solution contains 0.3 mg of chloride

G-2.2 PROCEDURE

G-2.2.1 Weigh 2.0 g test sample in 100 ml volumetric flask, dissolve the sample in methanol and make up to the mark. Transfer 5 ml of this solution into Nessler cylinder, add 2 ml nitric acid and make the volume to the mark with methanol, add 1 ml silver nitrate and mix well. Carry out a control test with 1 ml of standard chloride solution in place of test sample. Allow the cylinders to stand in dark for 5 min and then compare the opalescence of the solutions. The opalescence produced in the test sample shall not be greater than that produced in the control test.

G-3 METHOD 2

G-3.1 Reagents

G-3.1.1 *Concentrated Nitric Acid*

G-3.1.2 *Standard Silver Nitrate Solution* — 0.1 N

G-3.1.3 *Nitrobenzene*

G-3.1.4 *Standard Ammonium Thiocyanate Solution* — 0.1 N

G-3.1.5 *Ferric Ammonium Sulphate Indicator* — Approximately 5 percent

G-3.2 Procedure

Weigh accurately about 20 g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator.

G-3.3 Calculation

$$\text{Chloride(as Cl), percent by mass} = \frac{3.55\,(10\,N_1 - V\,N_2)}{M}$$

where,

N_1 = normality of standard silver nitrate solution,

V = volume in ml of standard ammonium thiocyanate solution consumed in the titration

N_2 = normality of the standard ammonium thiocyanate solution, and

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

ANNEX H

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

TEST FOR COPPER

H-1 GENERAL

Two methods have been prescribed for determination of copper in pyridine. Both the methods can be used for determination of copper on routine basis. However, in case of dispute, method 1 shall be treated as a referee method.

H-2 METHOD 1

H-2.1 Reagents

H-2.1.1 *Ascorbic Acid*

H-2.1.2 *Dilute Hydrochloric Acid*

H-2.1.3 *Sodium Acetate* — 40 percent (*m/v*)

H-2.1.4 *Bathocuproine Reagent* — Dissolve 0.1 g of Bathocuproine, sulfonated sodium salt in water and dilute to 100 ml.

H-2.2 Procedure

H-2.2.1 This test requires the residue obtained after evaporation as described in Annex-D from a 200 g of sample. Dissolve the residue in 1 ml of hydrochloric acid and about 10 ml of water, heat to boiling, and cool. Filter, if necessary, through pre-washed Whatman filter paper, No. 1, or equivalent, then wash dish with 10 ml of hydrochloric acid, adding washing to sample. Dilute to 100 ml with water, mix well, and transfer 1.0 ml (2 g) to a 50 ml beaker. Add 20 ml of hydrochloric acid.

Prepare a copper standard by dispensing 1.0 ml of 0.1 ml/ml of copper standard solution into a Nessler cylinder and adding 20 ml of hydrochloric acid. Using a pH meter adjust the pH to 7 to 8 with 10 percent ammonium hydroxide. Add 0.1 g of ascorbic acid, 2.5 ml of 40 percent (*w/v*) sodium acetate, and 0.5 ml of bathocuproine indicator, mix and observe after 1 minute. Any yellow-orange colour in the sample must not exceed that of the standard (5 ppm).

H-3 METHOD 2

H-3.1 Apparatus

H-3.1.1 *Nessler Cylinders — 50 ml capacity*

H -3. 2 Reagents

H - 3. 2.1 *Concentrated Hydrochloric Acid*

H -3.2.2 *Concentrated Nitric Acid*

H -3.2.3 *Citric Acid*

H-3.2.4 *Dilute Ammonium Hydroxide — A*approximately 5 N

H-3.2.5 *Sodium Diethyldithiocarbamate Solution —* Dissolve 1.0 g of sodium diethyldithiocarbamate in 1 000 ml of copper free water. Filter and keep in an amber bottle and protect from strong light.

H-3.2.6 *Standard Copper Solution* — Dissolve 0.3928 g of copper sulphate pentahydrate in copper free water and make up the volume to 1 000 ml. Take 100 ml of this solution and dilute again to 1 000 ml. One milliltre of the diluted solution contains 0.01 mg of copper (as Cu).

H-3.2.7 *Chloroform*

H-3.3 Procedure

H-3.3.1 Weigh 6.000 g of the material and dissolve it in about 50 ml of water. Neutralize with concentrated hydrochloric acid and add 4 to 5 drops of concentrated nitric acid. Boil and cool. Add 1 g of citric acid and adjust the pH to 9 by adding dilute ammonium hydroxide. Add 10 ml of sodium diethyldithiocarbamate solution and extract the yellow colour produced four times with 2.5 ml portions of chloroform. Collect the chloroform extracts and filter into a Nessler cylinder. Carry out a control test using 3 ml of standard copper solution in place of the material.

H-3.3.2 The material shall be considered not to have exceeded the limit prescribed in Table 1 if the intensity of the colour produced with the material is not greater than that produced in the control test.

ANNEX J

[*Table* 1, *Sl* (viii)]

TEST FOR OXIDIZABLE SUBSTANCES

J-1 REAGENTS

J-1.1 Standard Potassium Permanganate Solution — 0.1N

J-2 PROCEDURE

J-2.1 Take 5 ml of the material in a test tube and add 0.5 ml of standard potassium permanganate solution and shake it thoroughly.

J-2.2 The material shall be taken to have passed the test if the pink colour produced does not entirely discharge in 30 min.

ANNEX K

[*Table* 1, *Sl No* (ix)]

TEST FOR SULPHATES

K-1 APPARATUS

K-1.1 One Mark Graduated Flasks — 100 ml and 1 000 ml (*see* IS 915).

K- 1.2 Nessler Cylinders — 50 ml (*see* IS 4161)

K-2 REAGENTS

K-2.1 Denatured Spirit

K-2.2 Dilute Hydrochloric Acid — 5N (approximately)

K-2.3 Barium Chloride ($BaCl_2.2H_2O$) Solution — 10 percent (*m/v*)

K-2.4 Standard Sulphate Solution — Dissolve 1.48 g of ignited sodium sulphate (Na_2SO_4) in water. Transfer quantitatively to the 1 000 ml one mark graduated flask and make up the volume with water to the mark. Pipette 10 ml of this solution into the 100 ml one mark graduated flask and make up the volume up to the mark. One millimeter of this solution contains 0.1 mg of sulphates (as SO_4).

K-3 PROCEDURE

K-3.1 Dissolve 10.0 g of the material in 40 ml of water and transfer quantitatively into one of the Nessler cylinders. Add 10 ml of denatured spirit and 1 ml of hydrochloric acid; mix and add 1 ml of barium chloride solution. Allow to stand for 1 h. In another Nessler cylinder carry out a control test under the same conditions using 1 ml of standard sulphate solution, 37 ml water, 10 ml of denatured spirit, 1 ml of dilute hydrochloric acid, and 1 ml of barium chloride solution.

K-3.2 The material shall be taken to have not exceeded the limit prescribed for grade 1 if the turbidity produced with the material is not greater than that produced in the control test.

ANNEX L

[*Table* 1, *Sl No* (x)]

DETERMINATION OF MOISTURE CONTENT

L-1 GENERAL

Moisture is determined by the Karl Fischer method.

L-2 PROCEDURE

1 Weigh accurately 20 g of the material and determine the moisture content by the procedure given in IS 2362.

ANNEX M

(*Clause* 7)

SAMPLING OF PYRIDINE

M-1 GENERAL REQUIREMENTS FOR SAMPLING

M-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

M-1.2 The sampling instrument shall be clean and dry.

M-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.

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

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

M-1.6 The sample containers shall be of such a size that they are almost three-fourth filled by the sample.

M-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under **6.2**.

M-2 SCALE OF SAMPLING

M-2.1 Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

M-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

M-2.3 The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 4.

Table 4 Number of Containers to be Selected from Lots of Different Sizes

(*Clause* M-2.3)

Lot Size '*N*'	Sample Size '*n*'
(1)	(2)
3 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 and above	9

M-2.4 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS 4905 for guidance to random selection procedures.

M-3 TEST SAMPLE AND REFEREE SAMPLE

M-3.1 From each of the containers selected as in **M-2.3**, draw with the help of a sampling bottle a representative portion of the material from different parts of the container. Out of this portion from each container equal quantity of the material shall be taken and thoroughly mixed to form a composite sample of about 1 500 ml. This composite sample shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

M-3.2 The remaining portion corresponding to each of the selected containers as in **M-2.3** shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

M-3.3 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **M-1.7**.

M-3.4 The referee samples consisting of a composite sample and a set of *n* individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

M-4 TESTS

M-4.1 Tests for pyridine content and moisture shall be conducted on individual samples.

M-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

M-5 CRITERIA FOR CONFORMITY

M-5.1 For Individual Samples

The lot shall be declared as conforming to the requirements of pyridine content and moisture if each of the test results on the individual samples satisfies the corresponding requirement of the test.

M-5.2 For Composite Sample

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the relevant requirements given in **4** and Table 1.

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