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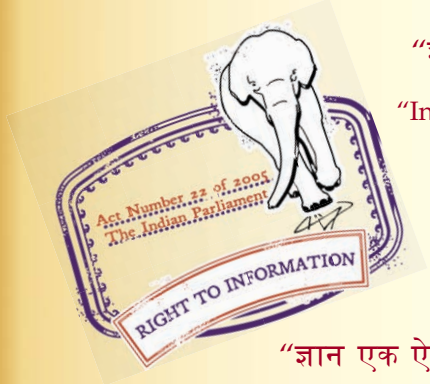
IS 7661 (1975): 4-aminoazobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS : 7661 - 1975
(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR 4-AMINOAZOBENZENE

UDC 667·21 : 547·556·33

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

Price Rs 7·00

July 1975

Indian Standard

SPECIFICATION FOR 4-AMINOAZOBENZENE

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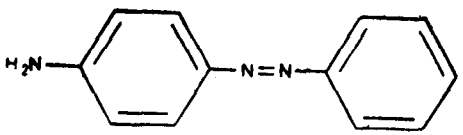
Indian Standard

SPECIFICATION FOR 4-AMINOAZOBENZENE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 6 May 1975, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 4-Aminoazobenzene ($C_{12}H_{11}N_3$) is an important starting material for many diazo dyes. It is also known as 4-benzene azoaniline. It has the following structural formula:



4-AMINOAZOBENZENE

(Molecular Mass 197·1)

0.3 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.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 4-aminoazobenzene.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of yellowish to reddish orange powder and free from visible impurities.

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

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4-AMINOAZOBENZENE

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	90	A-1
ii)	Aniline content, percent by mass, *Max*	0·2	A-2
iii)	2-Aminoazobenzene content, percent by mass, *Max*	2	A-3
iv)	Insolubles in diazo solution, percent by mass, *Max* (on 100 percent basis)	5	A-4
v)	Melting point, *Min*	119°C	A-5

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

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

c) Batch number;

d) Gross, net and tare mass; and

e) The minimum cautionary notice worded as under:

'POISONOUS ! AVOID INHALATION AND CONTACT WITH SKIN ! REPEATED ABSORPTION MAY RESULT IN BLADDER TUMORS .'

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 requiremen s of that standard under a well-defined system of inspection, testing and quality control

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

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, maybe obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples separately.

4.2.2 Tests for the determination of remaining characteristics, namely, aniline content , 2-aminoazobenzene content, insolubles in diazo solution, and melting point shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — 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.

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF TEST FOR 4-AMINOAZOBENZENE

A-1. ASSAY

A-1.0 The sample shall be assayed by either of the two methods given in **A-1.1** and **A-1.2**.

*Methods of sampling and tests for dye intermediates.
†Specification for water, distilled quality (*revised*).

A-1.1 Assay by Diazotization

A-1.1.0 *Outline of the Method* — Direct titration involving diazotization of the amine under acidic conditions with standard sodium nitrite solution using starch and potassium iodide test papers as external indicator.

A-1.1.1 *Reagents*

A-1.1.1.1 *Glacial acetic acid* — *See* IS : 695-1967*.

A-1.1.1.2 *Concentrated hydrochloric acid* — *See* IS : 265-1962†.

A-1.1.1.3 *Potassium bromide* — solid.

A-1.1.1.4 *Standard sodium nitrite solution* — 1 N.

A-1.1.1.5 *Starch and potassium iodide test papers*

A-1.1.2 *Procedure* — Dissolve 5·000 0 g of the sample in 200 ml of glacial acetic acid in a 500-ml conical flask. Heat moderately, if necessary. Cool the contents of the flask externally to 20°C and thereafter transfer the solution to a 2-litre beaker. Rinse the flask thoroughly with glacial acetic acid. Add 25 ml of concentrated hydrochloric acid, 500 ml of water and 5 g of potassium bromide. While stirring mechanically, titrate dropwise at about 20°C with standard sodium nitrite solution. (By keeping the solution for a longer time, without addition of sodium nitrite, the material will start crystallizing which should be avoided.) When about 24 ml of nitrite solution (corresponding to about 95 percent material) has been added, start testing on starch and potassium iodide test papers. As the end point is approached, add nitrite in 0·1 ml and finally in 0·05 ml increments. The end point is reached when an immediate faint blue-coloured ring appears, which can be obtained repeatedly during the period of 10 minutes without further addition of nitrite solution. Record the volume of titre used as V. Preserve the diazo solution obtained for the test in **A-4.**

A-1.1.3 *Calculation*

$$\text{Total amine, percent by mass} = \frac{V \times N \times 19{\cdot}71}{M} \quad \text{(let this value be } D\text{)}$$

where

V = volume in ml of sodium nitrite solution required for the sample,

N = normality of sodium nitrite solution, and

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

Specification for acetic acid ((first revision)*).

†Specification for hydrochloric acid (*revised*).

A-1.1.4 *Reporting* — Total amine content determined shall be corrected by the amount of aniline determined under **A-2**, when reporting the results:

Total amine content (excluding aniline), calculated from diazotization value, percent by mass $= D - (E \times 2{\cdot}12)$

where

D = total amine content inclusive of aniline (*see* **A-1.1.3**), percent by mass (on molecular mass 197·1); and

E = aniline content (*see* **A-2.3.2**), percent by mass (on molecular mass 93·12).

A-1.2 Assay by Chromatographic Method

A-1.2.0 *Outline of the Method* — Assay is done by column chromatographic method. The material is separated chromatographically and determined quantitatively by spectrophotometer.

A-1.2.1 *Apparatus*

A-1.2.1.1 *Chromatographic column*

A-1.2.1.2 *Spectrophotometer* — capable of reading at 370 mμ.

A-1.2.2 *Reagents*

A-1.2.2.1 *Toluene* — distilled.

A-1.2.2.2 *Alumina* — neutral.

A-1.2.2.3 *4-Aminoazobenzene* — pure.

A-1.2.3 *Procedure*

A-1.2.3.1 Weigh accurately about 50 mg of the sample. Transfer the sample to a dry 250-ml flask. To the flask, add about 200 ml of toluene and reflux for half an hour. Cool the solution to room temperature. Transfer the solution to a dry 250-ml volumetric flask. Wash the flask thoroughly with solvent and transfer the washings also to the volumetric flask. Finally dilute the solution to the mark and shake well.

Prepare an alumina column, using standard neutral alumina and toluene. The height of the alumina column shall be about 20 cm. Load 5 ml of the solution. Allow the solution to come down and when the

cotton on the alumina is just dry, add about 1 to 2 ml of toluene at a time till the cotton is free of colour. Then fill the column with toluene. Following bands are observed from bottom to top:

a) Very faint yellow,

b) Second very faint yellow, and

c) Main yellow band.

Collect the main yellow band in a dry 250-ml volumetric flask. Make up to the mark, shake well and take reading on a spectrophotometer at 370 mμ.

A-1.2.4 *Calculation* — Calculate the purity by comparing with the reference sample as follows:

$$\text{Purity, percent by mass} = \frac{D \times k \times d}{M} \times 100$$

where

D = optical density of the sample taken for the test,

k = constant (reciprocal of the optical density of 1 mg/100 ml of the reference sample),

d = dilution factor, and

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

A-2. DETERMINATION OF ANILINE CONTENT

A-2.0 Outline of the Method — After slurrying a known mass of the material with water, aniline is steam-distilled into dilute hydrochloric acid. The distillate containing aniline is diazotized with standard sodium nitrite solution.

A-2.1 Apparatus

A-2.1.1 *Distillation Flask* — 3 litres capacity.

A-2.1.2 *Condenser* — of not less than 45 cm length.

A-2.2 Reagents

A-2.2.1 *Hydrochloric Acid* — dilute (1 : 1).

A-2.2.2 *Concentrated Hydrochloric Acid* — *See* IS : 265-1962*.

A-2.2.3 *Standard Sodium Nitrite Solution* — 0·5 N.

A-2.2.4 *Starch and Potassium Iodide Test Papers*

*Specification for hydrochloric acid (*revised*).

A-2.3 Procedure — Place 100 g of the sample in a distillation flask and slurry with 750 ml of water. Connect the side arm of the distillation flask with the condenser. Steam distil the aniline while heating the flask from below. Collect the distillate in a 1-litre conical flask containing 100 ml of dilute hydrochloric acid. When about 500 ml of the distillate has been collected, stop distillation and cool the distillate to about 5°C by external cooling. Filter the distillate through filter paper (Whatman No. 1 or equivalent) and estimate aniline in the filtrate by diazotization.

A-2.3.1 *Diazotization* — Transfer the filtrate quantitatively to a 1-litre beaker. Add 25 ml of concentrated hydrochloric acid and 10 g of potassium bromide. Cool the beaker and the contents to 0 to 2°C in a suitable ice-bath and add a little crushed ice to the beaker. While continuously stirring the contents of the beaker, titrate at 0 to 2°C with standard sodium nitrite solution, adding the nitrite solution dropwise through a long stem funnel which dips into the solution. As the end point is approached, remove the long stem funnel, wash it thoroughly into the beaker, and add the nitrite solution, with the tip of the burette well under the surface of the solution, in 0·1 ml and finally in 0·05 ml increments. The end point is reached when an immediate faint blue-coloured ring appears which can be obtained repeatedly during the period of 10 min without further addition of nitrite solution. Record the volume of titre used as V.

A-2.3.2 *Calculation*

$$\text{Aniline, percent by mass} = \frac{(V - 0{\cdot}4) \times N \times 9{\cdot}3}{M}$$

where

V = volume in ml of standard sodium nitrite solution used,

N = normality of sodium nitrite solution,

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

0·4 = the correction factor for the dissolved 4-aminoazobenzene.

A-3. DETERMINATION OF 2-AMINOAZOBENZENE CONTENT

A-3.0 Outline of the Method — 4-Aminoazobenzene and 2-aminoazobenzene on reduction with either stannous chloride or zinc and hydrochloric acid, form aniline and *p*-phenylenediamine and aniline and *o*-phenylenediamine respectively. The *p*-phenylenediamine and *o*-phenylenediamine can be distinguished by their behaviour towards the reagent phenanthraquinone. The former does not react, while the latter forms bright yellow precipitate of diphenylene quinoxaline ($C_{20}H_{12}N_2$).

A-3.1 Reagents

A-3.1.1 *Zinc Dust*

A-3.1.2 *Glacial Acetic Acid*

A-3.1.3 *Acetic Acid Solution* — 30 percent (*v/v*).

A-3.1.4 *o-Phenylenediamine*

A-3.1.5 *Hydrochloric Acid* — 30 percent (*m/v*).

A-3.1.6 *Sodium Bisulphite Solution* — 20 percent (*m/v*).

A-3.1.7 *Phenanthraquinone-Bisulphite Solution* — Dissolve 10 g of phenanthraquinone in 100 ml of glacial acetic acid, heating, if necessary. Pour the clear solution in 1 000 ml of water. The material falls out in a fine state of division. Filter the material and wash with water. Take this already prepared phenanthraquinone in a small dish, add 20 ml of sodium bisulphite solution or for 10 g take 4 g of sodium bisulphite and dissolve in 20 ml of water. The contents of the dish will set soon to an almost white mass. Dissolve the mass in water and dilute to approximately 2 000 ml and filter.

A-3.1.8 *Sodium Acetate* — crystalline.

A-3.2 Procedure

A-3.2.1 Suspend 10 g of 4-aminoazobenzene in 150 ml of hydrochloric acid and 1 000 ml of hot water. Stir the solution while moderately heating over a burner. Reduce with sufficient quantity of zinc dust, adding in portions. To the decolourized solution add 150 g of crystalline sodium acetate. Allow it to cool thoroughly, filter and wash with water. Dilute to a known volume (*V*). *o*-Phenylenediamine is precipitated in the filtrate as follows:

> Mix 100 ml (or a suitable aliquot, according to the contents of *o*-phenylenediamine) of the prepared and filtered solution with a few drops of sodium bisulphite solution and 5 ml of 30 percent acetic acid in a conical flask. Dilute with water to about 500 ml. Precipitate the *o*-phenylenediamine with phenanthraquinone-bisulphite solution.

A-3.2.1.1 The precipitation of *o*-phenylenediamine with phenanthraquinone-bisulphite is to be followed under certain conditions because, if too large excess of the reagent is used for the precipitation of *o*-phenylenediamine, too high results are obtained. Besides, the reaction between phenanthraquinone and *o*-phenylenediamine should run slowly and proportionately. On this ground, the required quantity of the

reagent for the precipitation should be ascertained carefully as far as possible. In the best way one can proceed according to the following procedure:

First add only 20 ml of phenanthraquinone-bisulphite solution to the aliquot taken. Heat to boil; and boil for 5 min more. Filter a part of the reaction mixture. Divide the filtrate in two parts. One part is mixed with 3 drops of phenanthraquinone-bisulphite solution and the other part is mixed with several millilitres of aqueous solution of *o*-phenylenediamine. Both the solutions are heated to boil simultaneously and boiled for a little more time. Then acidify with few drops of hydrochloric acid. A sufficient quantity of phenanthraquinone-bisulphite is present if the turbidity obtained with *o*-phenylenediamine solution is stronger than that obtained with phenanthraquinone-bisulphite solution. Observe the turbidity only after heating the solution to boil. There may be maximum turbidity with phenanthraquinone-bisulphite solution, but it should not be a flocky separation. Turbidity produced on cooling is not to be considered.

If it is found that phenanthraquinone-bisulphite is present in excess, boil the main reaction mixture for 5 min without further addition of the reagent and repeat the test as stated above. If the test still detects excess phenanthraquinone, it can be assumed that sufficient quantity of the reagent has been added.

However, if the test shows the presence of *o*-phenylenediamine, another 20 ml of phenanthraquinone-bisulphite solution should be added to the main reaction mixture, boiled for 5 to 10 min and tested as above for the presence of excess of the reagent.

A-3.2.2 By this way if the quantity of phenanthraquinone-bisulphite solution adequate for complete precipitation of *o*-phenylenediamine has been ascertained, the original reaction mixture is further heated for 1 h in boiling water-bath for completion of conversion. Filter the precipitated condensation product through a folded filter paper. Transfer the precipitate to a beaker by means of 1 percent hydrochloric acid. Boil for some time. Filter the precipitate through a tared G-4 sintered glass or Gooch crucible. Dry at 100 to 110°C to constant mass. Cool in a desiccator and weigh.

A-3.2.3 If ascertaining the quantity of phenanthraquinone needs more partial precipitation, the condensation product is coloured dark very strongly. So the first test should be considered as a preliminary experiment. Carry out the final experiment by adding the quantity of phenanthraquinone-bisulphite solution (ascertained in the preliminary experiment), taking fresh aliquot of the solution.

A-3.3 Calculation

$$\text{2-Aminoazobenzene, percent by mass} = \frac{A \times V \times 70{\cdot}354\,5}{b \times M}$$

where

A = mass in g of the condensation product obtained,

V = volume in ml made after reducing the compound,

b = volume in ml of the aliquot, and

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

A-4. DETERMINATION OF INSOLUBLES IN DIAZO SOLUTION

A-4.1 Procedure — Observe the clarity of the diazo solution obtained in **A-1.1.2**. If more than traces of undiazotizable matter remain, filter the solution through a tared G-3 sintered glass crucible, wash thoroughly with water till free from chlorides. Dry the crucible with its contents at 100°C to constant mass. Cool in a desiccator and weigh.

A-4.2 Calculation

$$\text{Insolubles in diazo solution, percent by mass (on 100 percent basis)} = \frac{S \times 100}{M} \times \frac{100}{D}$$

where

S = mass in g of the residue,

M = mass in g of the material taken for diazotization in **A-1.1.2**, and

D = purity of 4-aminoazobenzene (*see* **A-1.1.4**).

A-5. DETERMINATION OF MELTING POINT

A-5.1 Determine the melting point of the material as prescribed in **8** of IS : 5299-1969*.

*Methods of sampling and tests for dye intermediates.

INDIAN STANDARDS

ON

DYE INTERMEDIATES

IS :

2630-1973 Nitrobenzene (*first revision*)
2740-1973 Sulphanilic acid, technical (*first revision*)
2741-1973 β-Naphthol (*first revision*)
2744-1964 α-Naphthylamine
2833-1973 Aniline, technical (*first revision*)
3229-1973 Naphthionic acid (sodium salt) (*first revision*)
3242-1965 β-Oxynaphthoic acid (bon acid)
3562-1965 *p*-Nitrotoluene, technical
4265-1967 4, 4'-Diaminostilbene-2, 2'-disulphonic acid
4334-1967 *o*-Chloroaniline
4335-1967 *m*-Chloroaniline
4336-1967 *p*-Chloroaniline
4425-1967 *p*-Nitrotoluene-*o*-sulphonic acid
4523-1968 Acetoacetanilide
4524-1960 Acetoacet-*o*-chloroanilide
4525-1968 *p*-Aminoacetanilide
4526-1968 2, 5-Dichloroaniline
4527-1968 2-Nitro-4-chlorotoluene
4528-1968 4, 4'-Dinitrostilbene-2, 2'-disulphonic acid (disodium salt)
5042-1969 1-Aminoanthraquinone
5043-1969 2-Aminoanthraquinone
5044-1969 Benzanthrone
5045-1969 Metanilic acid, technical
5299-1969 Methods of sampling and tests for dye intermediates
5438-1969 Nitrobenzene-*m*-sulphonic acid, sodium salt
5646-1970 *p*-Anisidine
5647-1970 *p*-Toluidine
5648-1970 *o*-Anisidine
5649-1970 *o*-Toluidine
6258-1970 *o*-Nitroanisole
6259-1971 Anthraquinone, technical
6260-1971 *p*-Nitroanisole
6264-1971 J-acid
6265-1971 Quinizarine, technical
6266-1971 1, 4-Diaminoanthraquinone, technical
6961-1973 3-Bromobenzanthrone, technical
6962-1973 3,9-Dibromobenzanthrone, technical

IS :

6977-1973	1, 5-Diaminoanthraquinone, technical
7359-1974	1-Chloroanthraquinone, technical
7360-1974	1, 5-Dichloroanthraquinone, technical
7362-1974	Tobias acid
7364-1974	*m*-Nitro-*p*-toluidine
7635-1975	2-Nitroaniline
7636-1975	4-Chloro-2-toluidine
7637-1975	5-Chloro-2-toluidine
7641-1975	4-Chloro-2-anisidine
7642-1975	4-Aminophenol
7643-1975	4-Chloro-2-nitroaniline
7644-1975	3-Nitroaniline
7645-1975	Phenyl J-acid, technical
7646-1975	Benzoyl J-acid, technical
7647-1975	Acetoacet-2-toluidine
7661-1975	4-Aminoazobenzene
7686-1975	3 (N. N-diethyl) aminophenol

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INDIAN STANDARDS

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Chemicals, inorganic (miscellaneous)
Chemicals, organic (miscellaneous)
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AMENDMENT NO. 1 NOVEMBER 2003
TO
IS 7661 : 1975 SPECIFICATION FOR 4-AMINOAZOBENZENE

[*Page* 3, *Foreword*, *Structural formula*] — Insert '(CAS No. 60-09-3)' at the end of structural formula.

[*Page* 4, *Table* 1, *Sl No.* (ii) *and* (iii), *col* 4] — Insert 'A-6' as an alternate method.

(*Page* 12, *clause* **A-5.1**) — Insert the following text after **A-5.1**:

A-6 THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

A-6.1 General

Impurities are determined by thin layer chromatography. Reference may be made to 'IS 5299 : 2001 Methods of sampling and tests for dye intermediates' for details of TLC test method to be followed. However, necessary details of test conditions are given here for guidance only.

1.	Product name	:	4-Aminoazobenzene
2.	Sample solution (on 100% basis)	:	2% in acetone
3.	Application/volume for spotting	:	10 µl for sample and 2 µl and 4 µl for impurities
4.	Standard	:	Reference standard
5.	Test substance for impurities	:	1) 2-Aminoazobenzene 2) Aniline (0.05% Solution in acetone)
6.	Plate type	:	Silica gel G
7.	Eluent	:	Toluene : Carbontetrachloride 50 : 50 (Ammonia Atmosphere)
8.	Elution time	:	40 min
9.	Temperature	:	25 ± 5°C

10. Detection spray	:	* PDAB solution	
11. Evaluation	:	Semi quantitative	
12. Approx Rf value — Main band	:	4-Aminoazobenzene	Rf 0.3
—Impurities		Aniline :	Rf 0.2
		2-aminoazobenzene :	Rf 0.4

*PDAB solution : *p*-Dimethylamino benzaldehyde
1 % solution in (1:0.5:0.5)
Methanol : Water : 5 N HCl

(PCD 11)

Reprography Unit, BIS, New Delhi, India