

भारतीय मानक
Indian Standard

IS 2833 : 2019

एनिलिन, तकनीकी — विशिष्टि
(दूसरा पुनरीक्षण)

Aniline, Technical — Specification
(*Second Revision*)

ICS 71.080.80

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March 2019

Price Group 3

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Aniline (C₆H₇N) finds extensive use in the dye industry. It is the starting material for many azo dyes. It is used in the manufacture of other intermediates as acetanilide, acetoacetanilide, aminoazobenene, methyl aniline, diethyl aniline, and dimethyl aniline, and many others.

This standard was originally published in 1964 and subsequently revised in 1973. In the first revision, modifications of almost all the requirements had been necessitated and maximum moisture content was specified. Newer techniques of analysis, such as polarography was used for the determination of nitrobenzene in aniline.

This revision has been undertaken with a view to making it appropriate to the current needs of the industry. In this revision, GC method for determination of assay percentage has been added and method for determination of nitrobenzene content has been updated. Test methods for relative density, crystallization point, distillation range and moisture content have also been updated.

The composition of the Committee, responsible for the formulation of this standard is given at Annex C.

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*)'. 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

ANILINE, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

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

2 REFERENCES

The following standards 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 agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
5299 : 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (<i>third revision</i>)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (<i>first revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)

3 REQUIREMENTS

3.1 Description — The material shall be a transparent oily liquid colourless to pale yellow when freshly distilled having a strong characteristic odour. It darkens on storage.

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable drums conforming to IS 2552.

4.2 Marking

4.2.1 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;
- c) Lot or batch number;
- d) Tare, net and-gross mass; and
- e) POISON [red label, printed on white background conforming to IS 1260 (Part I)].

Table 1 Requirements for Aniline, Technical
(Clause 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Cl No. in IS 5299 : 2001
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 27°C / 27°C	1.018 to 1.019	—	5
ii)	Crystallizing point, <i>Min</i>	-6.4°C	—	8
iii)	Distillation range	5 to 95 ml shall distil within the range of 1°C including 184°C	—	7
iv)	Assay, percent by nitrite value <i>Min</i>	99	A	—
v)	Assay, percent by GC, <i>Min</i>	99.5	B	—
vi)	Nitrobenzene content by GC, ppm <i>Max</i>	50	B	—
vi)	Moisture content, percent by mass, <i>Max</i>	0.3	—	10.2

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4.2.2 Each container shall, in addition, bear the minimum cautionary notice worded as under:

KEEP WELL CLOSED AND PROTECTED FROM LIGHT, TOXIC, AVOID CONTACT WITH SKIN

4.2.3 *BIS Certification Marking*

Each container may also be marked with the Standard Mark.

4.2.3.1 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 4 of IS 5299.

5.2 Number of Tests

5.2.1 Tests for the determination of crystallizing point and assay shall be conducted on each of the individual samples by using test methods prescribed under Table 1.

5.2.2 Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of crystallizing point, assay by nitrite value and assay by gas chromatography, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 QUALITY OF REAGENTS

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

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

ANNEX A

(Table 1)

ASSAY BY NITRITE VALUE

A-1 REAGENTS

A-1.1 Concentred Hydrochloric Acid

A-1.2 Potassium Bromide

A-1.3 Standard Sodium Nitrite Solution — 0.1 N

A-1.4 Starch Iodide Papers.

A-2 PROCEDURE

A-2.1 Weigh accurately about 0.40 g of the sample in watch glass transfer it to a 500 ml glass beaker. Add 200 ml of distilled water and 15 ml concentrated hydrochloric acid. Stir it on magnetic stirrer till solution is clear. Then cool it to 15°C by adding Ice. Add approximately 5 gm potassium bromide.

Titrate, while stirring mechanically, with sodium nitrite solution using starch iodide paper. The end point is reached when Blue coloured ring appears which can be obtained repeatedly for a period of 10 min without further addition of nitrite solution

A-3 CALCULATION

$$\text{A-3.1 Assay by Nitrite Value} = \frac{V \times N \times 93}{M \times 10}$$

Where,

V = Volume in ml, of sodium nitrite solution used;

N = Normality of sodium nitrite solution; and

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

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ANNEX B

(Table 1)

DETERMINATION OF ANILINE AND NITROBENZENE CONTENT BY GAS CHROMATOGRAPHY

B-0 PRINCIPLE OF GAS CHROMATOGRAPHY

The principle of Gas chromatography is the technique in which the components of a mixture are distributed between two phases one is stationary and one mobile. The mobile phase passes through the stationary phase, elutes the separated components one by one. The eluted components in the mobile phase are detected by a detector block and recorded by a [mv] recorder in terms of the variables of the components.

B-1 GC EQUIPMENT

Gas Chromatography system with Flame ionization detector and auto injector.

B-2 GC PARAMETERS

Column:	DB 1701 capillary column 30 Mtr × 0.25 mm × 1.0 μm
Or equivalent column	[Part No.122-0733]
Injector:	split Temperature: 275°C Split ratio: 30
Carrier gas:	Nitrogen or helium with flow control mode pressure
Carrier gas Flow rate	30 ml/min
Carrier gas pressure	100 kpa
Column Oven Temperature	100°C

Oven temperature:

Heating rate [°C/min]	Temperature [°C]	Hold Time [min]
-----	100	2
10	230	15

Run Time: 30 min

FID - Parameters

Hydrogen Flow:	40 ml/min
Air Flow:	400 ml/min
Temperature:	275°C

Remarks:

Aging of the column can influence the separation so that the conditions parameters like

Temperature and flow may have to be changed.

B-3 STANDARD AND SAMPLE PREPARATION

Use neat standard and sample for injection.

B-4 INJECTION VOLUME

Aniline standard and sample	1.0 μl
Nitrobenzene	0.1 μl

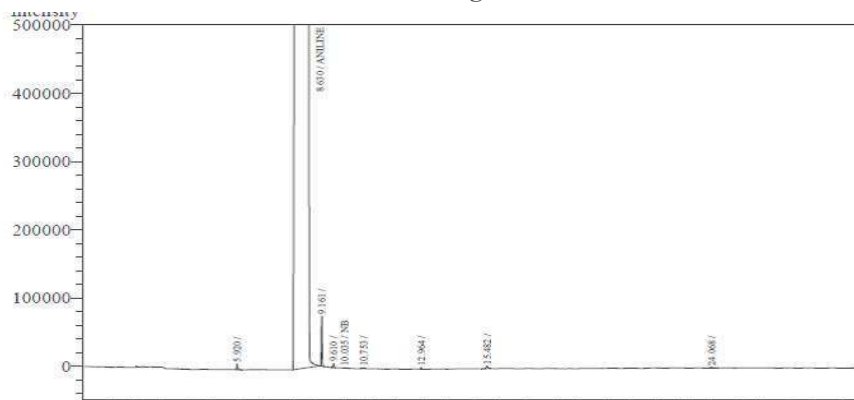
B-5 RETENTION TIME

Aniline	8.63 min
Nitro benzene	10.03 min

B-5 CALCULATION

Take value of aniline and nitrobenzene by area normalization.

Chromatogram



ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

<i>Organization</i>	<i>Representative(s)</i>
ICT, Mumbai	PROF P. M. BHATE, (<i>Chairman</i>)
Atul Ltd., Gujarat	DR M. U. RAHMAN DR J. G. DESAI (<i>Alternate</i>)
Aarti Industries Limited, Mumbai	SHRI KIRIT H. DESAI
Alkyl Amines Chemicals Ltd., Mumbai	SHRI S. V. NIKUMBHE SHRI KIRAT PATEL (<i>Alternate</i>)
Archroma, Thane	DR RAJESH RAMAMURTHY MR HEMANT MHADESHWAR (<i>Alternate</i>)
BASF India, Mumbai	SHRI UDAY KULKARNI
Central Revenues Control Laboratory, New Delhi	DR T. A. SREENIVASA RAO SHRI S. C. MATHUR (<i>Alternate</i>)
Colourtex Industries Limited, Mumbai	DR PANKAJ DESAI SHRI ISMAIL HABIBULLA (<i>Alternate</i>)
Deepak Nitrite Ltd, Vadodra	DR J. K. ASTIK
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	DR PARITI SIVA RAMA KUMAR
Gujarat Dyestuffs Manufacturers' Association (GDMA), Ahmedabad	SHRI R. S. PATEL SHRI BANSIBHAI PATAL (<i>Alternate</i>)
Gujarat Pollution Control Board(GPCB), Ghandhinagar, Ahmedabad	SHRI Y. A. TAI
Heubach, Colour Pvt. Ltd, Mumbai	SHRI J. I. SEVAK SHRI VINOD MADHUKAR (<i>Alternate</i>)
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
Jay Chemical Industries, Ahmedabad	SHRI VILPESH YADAV SMT MAITRI VYAS (<i>Alternate</i>)
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Ministry of Chemicals & Fertilizers, Department of Chemicals & Petrochemicals, New Delhi	SHRI JASBIR SINGH
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The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	SHRI V. R. KANETKAR
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Member Secretary

SHRI CHANDRAKESH SINGH
SCIENTIST C (PCD), BIS

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FOR REVISION OF INDIAN STANDARD**

(Foreword)

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Atul Ltd., Gujarat	DR. J. G. DESAI
Aarti Industries Limited, Mumbai	SHRI KIRIT H. DESAI
Heubach, Colour Pvt. Ltd, Mumbai	SHRI J. I. SEVAK
Jay Chemical Industries, Ahmedabad	SHRI P. S. KULKARNI

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Amendments Issued Since Publication

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