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भारतीय मानक मसौदा

ओरथो-एनीसिडीन — विशिष्टि

(IS 5648 *का दूसरा पुनरीक्षण*)

Draft *Indian Standard*

o-ANISIDINE –– SPECIFICATION

(*Second Revision of* IS 5648)

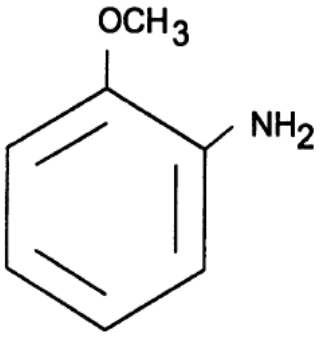
(ICS 71.080.60)

Dye Intermediates Sectional Committee, PCD 26	Last date for comment 27th September 2025

FOREWORD

(*Formal clauses to be added later*)

o-Anisidine is an important dye intermediate, used in the manufacture of dyes. It is represented by the following structural formula:



o-Anisidine
(Molecular Mass: 123.15)
CAS No. 90-04-0

This standard was first published in 1970 and subsequently revised in 2003 in light of experience gained during the long span of period. In this revision, the following major changes have been incorporated:

a. Determination of purity and impurities content by gas chromatography have been added.
b. Moisture content by Karl fischer has been added

c. Requirement for marking of pictograms, signal word, hazard statement, and precautionary statement as given in Annex D, which are derived from GHS guidelines have been incorporated. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated.

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.

1 SCOPE

This draft standard prescribes the requirements and methods of sampling and test for *o*-anisidine.

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 edition indicated was valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless to reddish liquid and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given under Table 1, when tested according to the methods prescribed col (4) and col (5) of Table 1.

Table 1 Requirements for *o*-Anisidine

(*Clauses* 3.2, 5.3 *and* 6.1)

SL No.	CHARACTERISTIC	REQUIREMENT	Method of test , Ref to	
			Annex	Clauses of IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Assay by nitrite value, percent by mass, Min	98.0	—	**13**
	Or			
	Purity, percent area by GC[1], Min	99.5		
ii)	Impurities by GC:			
	a) *p*-Anisidine, percent area by GC, Max	0.1		
	b) *m*-Anisidine, percent area by GC, Max	0.2	A	—
	c) *o*-chloroanisole, percent area by GC, Max	0.2		
	d) *o*-Chloroaniline, percent area by GC, Max	0.2		
	e) *o*-nitroanisole, percent area by GC, Max	0.05		
iii)	Matter insoluble in hydrochloric acid, percent by mass, Max	0.2	—	**11**
iv)	Moisture content by Karl Fischer, percent by mass, *Max*	0.50	B	—
v)	Crystallizing point[2], °C, *Min*	5.2	C	—

[1] In case of disputes, the sample shall satisfy the requirement of purity by GC, percent by area.
[2] Crystallizing point is an optional requirement. *o*-Anisidine is known to exist in more than one crystalline form and therefore, its crystallizing point determination is likely to give varying results. Hence it is necessary to heat the sample to its boiling point, cool it and then re-determine the crystallizing point.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or as agreed to between the purchaser and the supplier.

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 and his recognized trade-mark, if any;
c) Batch number;
d) Gross, net and tare mass;
e) Month and year of manufacturing;
f) Shelf life of the material; and
g) Pictograms, signal word, hazard statement and precautionary statement as mentioned in Annex D; and
h) Any other statutory requirement.

4.2.1 *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 shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

Test for the determination of all characteristics, shall be conducted on the composite sample.

5.3 Criteria for Conformity

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

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in col (4) and col (5) of Table 1.

6.2 Quality of Reagents

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, *Sl. No.* (ii)]
DETERMINATION OF PURITY AND IMPURITIES BY GAS CHROMATOGRAPHIC ANALYSIS

A-1 GENERAL

Determination of *o*-Anisidine (Purity) and impurities namely p-Anisidine, m-Anisidine, o-Chloroanisole, o-Chloroaniline and *o*-nitroanisole by Gas Chromatography instrument through area percent calculation.

A-2 APPARATUS

A-2.1 Analytical Balance

A-2.2 Volumetric Flask — 10 ml (Class A)

A-2.3 Pipette — 1 ml (Class A)

A-2.4 Micro syringe

A-2.5 Gas Chromatograph — Any gas chromatograph equipped with a Flame Ionization Detector (FID).

A-2.5.1*Column* — **(** 5%-Phenyl)-methylpolysiloxane with length 30m, inner diameter 0.320 mm and film thickness 0.25µm or equivalent.

A-2.5.2 *Gas Chromatography Parameters*:

Carrier Gas : Nitrogen

Injector Temperature : 250 °C

Column Oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	60	0
10	240	15

Hydrogen flow : 30 ml/min

Zero air flow : 300 ml/min

Purge Flow : 2.0 ml/min

Make up gas (N_2) flow : 25 ml/min

Split Ratio : 1 : 50

Detector Type : Flame Ionization Detector (FID)

Detector Temperature : 250 °C

Injection Volume : 0.2 µl

Total run time : 33 min

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-3 REAGENT

Methanol — Solvent

A-4 PROCEDURE

Take 1.0 g of *o*-anisidine and make up to 10 ml with methanol in volumetric flask. Now, dissolve properly and take 0.2 µl of sample as prepared micro syringe. Confirm there are no air bubbles in the syringe and inject the sample and allow the run to complete the run time.

A-5 PEAK TIME

	Peak time (min)
o- Anisidine	7.33
o-Chloroaniline	6.63
o-Chloroanisole	6.53
p-Anisidine	7.8
m-Anisidine	8.05
o-Nitroanisole	9.55

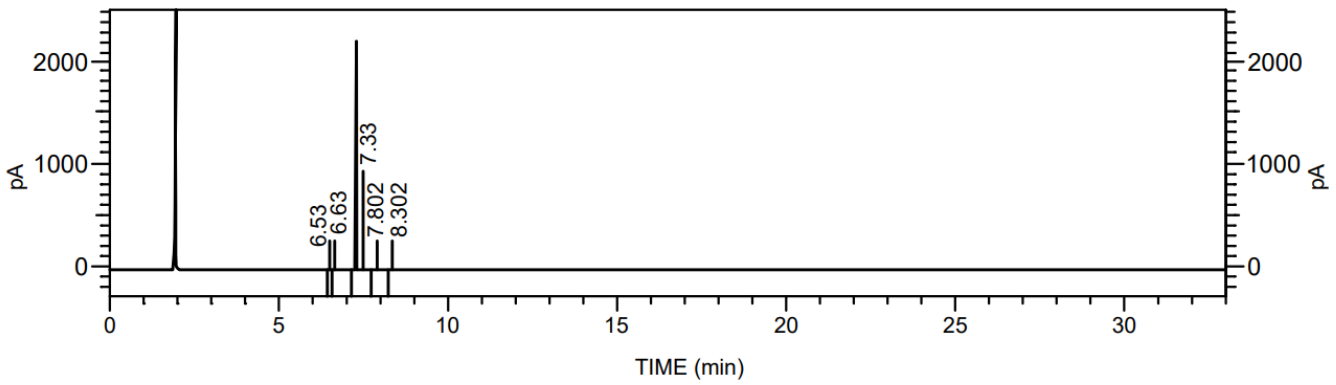


FIG.1 TYPICAL CHROMATOGRAM

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to *o*-Anisidine on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with standard *o*-Anisidine.

$$o\text{-Anisidine, percent by area} = \frac{p-\text{Anisidine } \textit{Peak area in the sample}}{\textit{Sum Areas of all peaks in the chromatogram}} \times 100$$

A-6.2 Similarly, *p*-Anisidine, *m*-Anisidine, *o*-Chloroanisole, *o*-Chloroaniline and *o*-nitroanisole content shall be calculated.

ANNEX B

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

DETERMINATION OF *O*- ANISIDINE MOISTURE CONTENT BY KARL FISCHER

B-1 APPARATUS

B-1.1 Karl Fischer Moisture Analyzer

B-1.2 Analytical Balance

B-1.3 Pipette — 1 ml (Class A)

B-2 REAGENTS

B-2.1 Karl Fischer reagent

B-2.2 Methanol Dried

B-3 PROCEDURE

Add approximately 25 ml to 30 ml methanol in titration vessel and stir with magnetic stirrer. Now, add Karl Fischer reagent to complete the neutralization of methanol. Now, enter sample details in the instrument. Pipette 1.0 ml of *o*-anisidine. Now, add the 1 ml sample in the titration vessel and press START to continue titration. Ensure proper and complete addition of sample in vessel. Once the sample is added, the instrument automatically starts addition of Karl

Fischer reagent in the titration vessel to titrate moisture present in sample. Instrument will stop adding Karl Fischer reagent automatically once it reaches the electrometric endpoint. Note down the burette reading.

B-4 CALCULATION

$$\text{Moisture content, percent w/w} = \frac{\text{B.R.} \times \text{Factor of KF} \times 100}{\text{Volume taken of sample} \times \text{Density} \times 1000}$$

where

B.R. = volume, in ml of karl fischer reagent consumed;

F = karl fischer reagent factor, in mg/ml;

V = Volume, in ml, of sample taken; and

D = Density (ρ =1.08)

ANNEX C
[*Table* 1, *Sl. No*. (v)]
DETERMINATION OF CRYSTALLIZATION POINT

C-1 APPARATUS

C-1.1 Thermometer

NOTE — The thermometer shall bear a calibration certificate from any institution authorized to issue certificate traceable to international or national measurement standards.

C-1.2 Dry Heating Block

C-1.3 Stainless steel Wire

C-1.4 Analytical Balance

C-2 PROCEDURE

Take approximately 15 g to 20 g of sample (*o*-Anisidine) in a test tube and heat the sample in a dry heater till it completely melts. Remove the test tube from the dry heating block and place thermometer in the test tube in such a way that the thermometer bulb is fully covered by sample material as shown in the figure below:

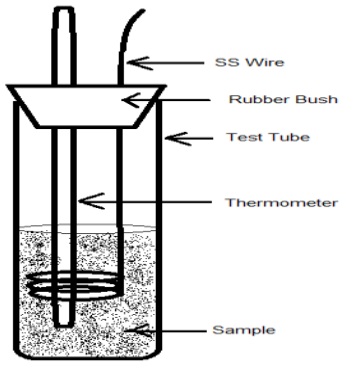


FIG. 2 APPARATUS

Now, stir the sample with stainless steel wire with vertical movement until solidification takes place. The constant temperature observed during solidification of the sample material shall be considered as crystallization point.

ANNEX D

[*Foreword and* 4.2.1 *(h)*]

Pictograms, signal word, hazard statement and precautionary statement:

Pictogram(s)		
Signal Word	**DANGER(ACUTE TOXIC)**	**HEALTH HAZARD**
Hazard Statement	**H301** Toxic if swallowed **H311** Toxic if swallowed **H331** Toxic if inhaled **H341** Suspected of causing genetic defects **H350** May cause cancer	
Precautionary Statement	**Precautionary statement(s) Prevention** **P201**: Obtain special instructions before use **P202**: Do not handle until all safety precautions have been read and understood. **P261**: Avoid breathing dust/fume/gas/mist/vapours/ spray **P262**: Do not get in eyes, on skin, or on clothing **P264**: Wash your hand thoroughly after handling. **P270**: Do not eat, drink or smoke when using this product. **P271**: Use only outdoors or in a well-ventilated area. **P272**: Contaminated work clothing should not be allowed out of the workplace. **P280**: Wear protective gloves/protective clothing/eye protection/face protection. **P281**: Use personal protective equipment as required. **Precautionary statement(s) Response** P301+P310: IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician. **P302+P352**: IF ON SKIN: Wash with soap and water. **P304 + P340** – IF INHALED: Remove person to fresh air and keep comfortable for breathing. **P308+P313**: IF exposed or concerned: Get medical advice/attention. **P311**: Call a POISON CENTER or doctor/physician.	

P312: Call a POISON CENTER or doctor/physician if you feel unwell.
P321: Specific treatment (see ... on this label).
P322: Specific measures (see ... on this label).
P330: Rinse mouth
P361: Remove/Take off immediately all contaminated clothing.
P362: Take off contaminated clothing and wash before reuse.
P363: Wash contaminated clothing before reuse.

Precautionary statement(s) Storage
P403+P233: Store in a well ventilated place. Keep container tightly closed.
P405 – "Store locked up."

Precautionary statement(s) Disposal
P501 Dispose of contents/container in accordance with local regulations.