BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

2-NITROANILINE - SPECFICATION

(*Second Revision of* IS 7635)

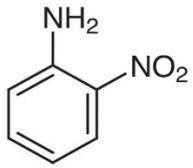
(ICS 71.080.30)

Dyes Intermediates Sectional Committee, PCD 26	Last date for comments 05 December 2023

FOREWORD

(*Formal clauses to be added later*)

2-Nitroaniline ($C_6H_6N_2O_2$) is an intermediate used in the manufacture of dyestuffs and pigments. It is also used as a diazo component in azoic dyeing in which case it is known as fast orange GR base. It is also known as 2-nitro-1-aminobenzene. It has the following structural formula:



2-Nitroaniline
Molecular Mass : 138
CAS Number : 88-74-4

This standard was originally published in 1975 and subsequently revised in 1992. In first revision, requirements of assay and crystallizing point were updated and requirement of impurities such as ortho-nitrochlorobenzene and para-nitroaniline were incorporated. In this (*second*) revision, requirement for determination of 2-nitroaniline and impurities such as ortho-nitrochlorobenzene and para-nitroaniline by gas chromatography have been incorporated. The new requirements of moisture content, acetone insoluble and melting point have also been incorporated and the requirement of crystallization point is deleted.

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as mentioned at Annex F**,** which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines were 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

1.1 This standard prescribes the requirements and the methods of sampling and testing for 2-nitroaniline.

2 REFERENCES

The following standards contain provisions which through reference in the 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 the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

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

3 REQUIREMENTS

3.1 Description

The material shall consist of orange to golden yellow crystalline needles, free from lumps and extraneous substances flakes.

3.2 The material shall also comply with the requirements as given in Table 1, when tested according to the methods prescribed col 4 of Table 1.

Table 1 Requirements for 2-Nitroaniline
(*Clause*s 5.3.1, 5.3.2 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Methods of Test, Ref to Annex**
(1)	(2)	(3)	(4)
i)	Assay by GC, percent by area, *Min*	99.00	A
	OR		
ii)	Assay (by titration) percent by mass, *Min*	98.50	B
iii)	Impurities		
	a) ortho-Nitrochlorobenzene content, *Max*	0.20	A
	b) para-Nitroaniline content, *Max*	0.20	
iv)	Moisture content by Karl Fischer, *Max*	0.50	C
v)	Acetone Insoluble , *Max*	0.20	D
vi)	Melting point[1)]	70 °C to 72 °C	E

[1)]Melting point is an optional parameter to be tested.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

4.2.1 Each container 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 manufacture;
f) Shelf life of the material; and
g) 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 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for assay by titration shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of all other characteristics under 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 requirement of assay by titration if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Samples*

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

6. TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 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.* (i), (iii)]

DETERMINATION ASSAY OF 2-NITROANILINE BY GAS CHROMATOGRAPHY

A-1 GENERAL

2-Nitroaniline and impurities like ortho-nitrochlorobenzene and para-nitroanline are determined using gas chromatography (GC).

A-2 APPARATUS

A-2.1 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID), a split/ splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

A-2.1.1 Gas Chromatography Parameters

Column : DB-1701 (30 m,0.25 mm ID,1.0 µm) or Equivalent

Carrier gas : Nitrogen

Injector temperature : 280 °C

Column oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	120	0.0
10	240	13

Nitrogen pressure : 110 kPa (16psi)

Hydrogen flow : 30 ml/min

Air flow : 300 ml/min

Column flow : 1.11 ml/min

Septum purge flow : 3.0 ml/min

Make up flow : 25 ml/min

Split ratio : 1:40

Detector type : FID

Detector temperature : 280 °C

Injection volume : 1.0 µl

Run time : 25.0 min

A-2.2 Analytical balance

A-2.3 Volumetric flask

A-2.4 Pipettes

A-2.5 Glass beaker

A-2.6 Sonicator

A-2.7 Water bath

A-3 REAGENTS

A-3.1 2- Nitroaniline

A-3.2 ortho-Nitrochlorobenzene

A-3.3 para-Nitroaniline

A-3.4 Acetone

A-4 PROCEDURE

A-4.1 Standard Solution preparation

Weigh accurately 1.0 g of the standard into a 10 ml volumetric flask, dissolve and dilute to volume with acetone.

A-4.2 Test sample preparation

Weigh accurately 1.0 g of the sample into a 10 ml volumetric flask, dissolve and dilute to volume with acetone.

NOTE — The weights and volumes given are the recommended amounts for routine quantitative analysis. Alternative amounts may be used, provided that the final concentrations remain the same.

A-5 PEAK TIME

2-Nitrochlorobenzene content	9.22 min
2-Nitroaniline	12.66 min
4-Nitroaniline content	18.38 min

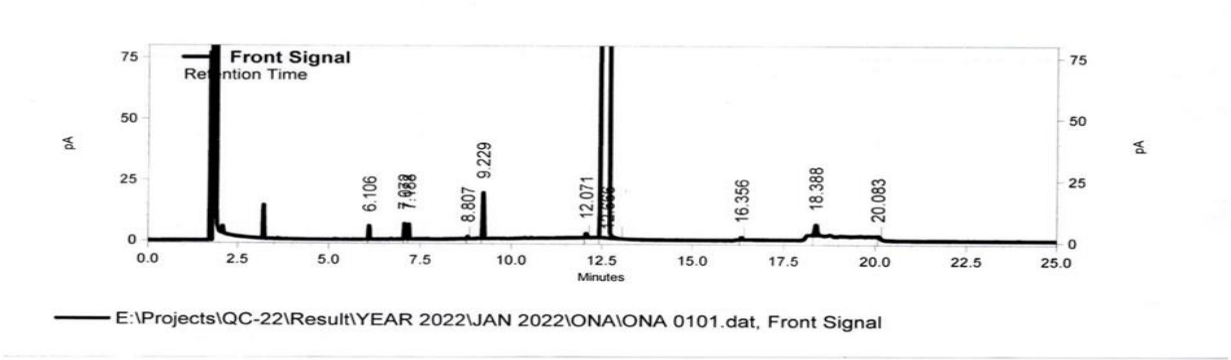


FIG. 1. TYPICAL CHROMATOGRAPH

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to 2-nitroaniline 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 2-nitro aniline.

$$\text{2-Nitroaniline, percent by area} = \frac{\text{2 Nitroaniline Peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

A-6.2 Similarly, contents of ortho-nitrochlorobenzene and para- nitroaniline shall be calculated.

ANNEX B
[*Table* 1, *Sl. No*. (ii)]
ASSAY (BY TITRATION) FOR 2- NITRO ANILINE

B-1 REAGENTS

B-1.1 Acetic acid

B-1.2 Concentrate Hydrochloric acid

B-1.3 Potassium bromide

B-1.4 Sodium nitrite Solution

B-2 PROCEDURE

Take 1g to 1.5 g sample on butter paper and transfer it in 1 litre beaker. Add 50 ml acetic acid. Stir with a glass rod and heat it up to dissolve, then add 30 ml concentrated hydrochloric acid (HCl) and then cool to room temperature. Now add 300 ml to 400 ml of distilled water. Then add washed clean ice pieces and maintain temperature 0 ºC to 5 ºC. Add 0.5 g potassium bromide (KBr) and titrate against 0.2 N sodium nitrite solution ($NaNO_2$). When observing the end point as a faint blue ring just appears on starch iodide paper, stop the addition and stir it for a few min. Now confirm again the end point on starch iodide paper and note down the burette reading.

B-3 CALCULATION

$$\text{Assay of 2-Nitroanline, percent by mass} = \frac{\text{V} \times \text{N} \times 138}{M}$$

where

V = Volume of standard sodium nitrite solution used, in ml;
N = Normality of the sodium nitrite solution; and
M = Mass of the sample taken for the test, in g.

ANNEX C
[*Table* 1, *Sl No.* (iv)]
DETERMINATION OF MOISTURE CONTENT BY KARL FISCHER

C-1 APPARATUS

C-1.1 Karl Fischer apparatus

C-1.2 Weighing Balance

C-2 REAGENTS

C-2.1 Methanol

C-2.2 2-Nitroaniline

C-3 PROCEDURE

Transfer about 40 ml of methanol into the titration vessel and titrate with the Karl Fischer reagent to the electrometric end point to consume any moisture that may be present. Quickly transfer about 0.5 g to 1.0 g sample into the titration vessel of Karl Fischer apparatus. Taking precaution that complete test substance is being transferred into the solvent and should not stick to the walls of the titration vessel. Mix thoroughly by stirring and titrate with Karl Fischer reagent to the end point under stirring.

C-4 CALCULATION

Calculate the moisture content of the sample by using the following formula

$$\text{Moisture content by Karl Fischer, by mass percent} = \frac{\text{KF reagent volume in ml X KF factor (mg/ml)} \times 100}{\text{Weight of the sample in gram} \times 1000}$$

ANNEX D
[Table 1 Sl No. (v)]
DETERMINATION OF ACETONE INSOLUBLE

D-1 APPARATUS

D-1.1 Weighing Balance

D-1.2 Filter Paper

D-2 PROCEDURE

Weigh filter paper using a balance with 0.0001 g precision. Record weight at initial weight of filter paper (I wt.). Connect assembly to vacuum pump. Take 5.0 g of the sample in approximately 100 ml acetone and dissolve the sample. Record weight of sample as (S wt.). Turn vacuum on and filter above solution. Wash the filter paper with 50 ml Acetone to dissolve the sample. Remove filter paper from filter assembly and dry at 80 ^{o}C for 1 h. Weigh the filter paper and record weight as final weight of paper (F wt.).

D-3 CALCULATION

$$\text{Percent insoluble} = \frac{F\,wt - I\,wt}{S\,wt} \times 100$$

where,

F wt. = Final weight of filter paper
I wt. = Initial weight of filter paper
S wt. =Weight of sample

ANNEX E
[Table 1 Sl No. (vi)]
DETERMINATION OF MELTING POINT

E-1 APPARATUS

E-1.1 Capillary tube

E-1.2 Melting Point Apparatus

E-1.3 Thermometer/ Temperature Controller

E-2 PROCEDURE

Grind the sample into powder form and Fill it in a one end sealed glass capillary tube. Keep this capillary tube in a melting point apparatus, start slowly heating and observe the temperature in a calibrated thermometer/temperature controller. When substance in the capillary tube starts melting, note down the temperature and when the substance is completely melted, note down the temperature

ANNEX F
(Forward)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :

Signal Word : **HEALTH HAZARD** **DANGER**

Hazard Statement :
H301 Toxic if swallowed.
H311 Toxic in contact with skin.
H412 Harmful to aquatic life with long lasting effects.
H373 May cause damage to organs through prolonged or repeated exposure.
H331 Toxic if inhaled.

Precautionary Statement :

Prevention
P260 do not breathe dost/ Hume/ gas/ mist/ vapours/ spray.
P270 do not eat, drink ok or smoke when using this product.
P271 use only outdoor or in a well ventilated area.
P280 where productive gloves/ protective clothing/ eye protection/ face protection.

Response
P301+P310 IF SWALLOWED: immediately call a POISON CENTER/ doctor/ physician/ first aider.
P330 Rinse mouth.
P302+P350 IF ON SKIN: wash with plenty of water and soap.
P304+P340 IF INHALED: remove person to fresh air and keep comfortable for breathing.

Storage
P403+P233 Store in a well ventilated place. keep container tightly closed.
P405 Store locked Up

Disposal
P501 Dispose of contents/ container in accordance with local regulations