IS 3229 (2003): Naphthionic Acid (Sodium Salt) [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

नैपथीयनिक एसीड (सोडियम साल्ट)—विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

NAPHTHIONIC ACID (SODIUM SALT)—SPECIFICATION

(Second Revision)

ICS 71.080.80

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2003

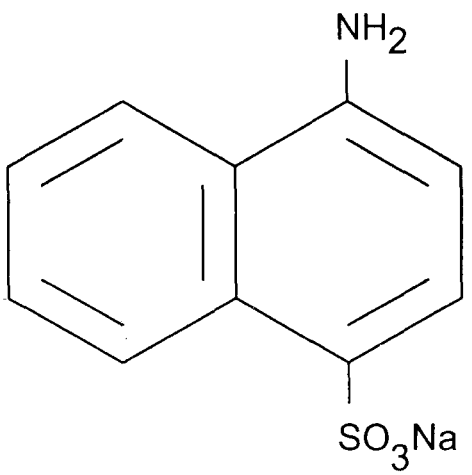
Price Group 3

FOREWORD

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

This standard was first revised in 1973. This revision is being carried out to upgrade the specification of naphthionic acid to align with the prevalent quality in the market. Stringent requirements for α-naphthylamine content and moisture content have been prescribed in this revision. Besides the complete testing has been recommended on the sample as such, deleting the preparation of sample for tests. Thin layer chromatography has been included as the method of test to keep pace with the present trends in the industry.

Naphthionic acid (sodium salt) ($C_{10}H_8O_3NS\,Na$), which is commonly known as sodium naphthionate and chemically named as 4-amino-1-naphthalene sulphonic acid, monosodium salt is an important dye intermediate and is widely used in the manufacture of azo dyes. It is represented by the following structural formula:



Naphthionic Acid (Sodium Salt)
(Molecular Mass 245.2)
[CAS NO. 130-13-2]

The composition of the Committee responsible for formulation of this standard is given in 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

NAPHTHIONIC ACID (SODIUM SALT) — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for naphthionic acid (sodium salt).

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 this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 2001	Methods for sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of pinkish-grey to grey powder when anhydrous. The hydrated product is pinkish crystalline and contains some lumps.

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers like jute bags or drums (*see* IS 2552) lined with polyethylene film or as agreed to between the supplier and the purchaser.

4.2 Marking

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

a) Name of the material;

b) Indication of the source of manufacture;

c) Lot or batch number;

d) Tare, gross and net mass;

e) Recognized trade-mark, if any; and

f) The minimum cautionary notice worded as under:

'IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS'.

4.2.1 *BIS Certification Marking*

4.2.1.1 Each container may also be marked with the Standard Mark.

4.2.1.2 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986

Table 1 Requirements for Naphthionic Acid (Sodium Salt)

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Clause of IS No.
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass exclusive α-naphthylamine, *Min* (calculated on molecular mass 245.2)	75	A	–
ii)	Impurities as α-naphthylamine content, *Max*, ppm	100	B	–
iii)	Matter insoluble in water, percent by mass, *Max*	0.1	–	11 of IS 5299

and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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

Tests for assay, α-naphthylamine content and matter insoluble in water shall be performed 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 this standard, if each of the individual test results satisfies the relevant requirements given at **3.1** and in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of matter insoluble in water and description while tested on the composite sample, the test result shall satisfy the relevant requirements given in Table 1 and **3.1**.

6 TEST METHODS

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

METHODS OF TEST FOR NAPHTHIONIC ACID (SODIUM SALT)

A-1 ASSAY DETERMINATION

A-1.0 Outline of the Method

The material is dissolved in aqueous alkaline solution. A known volume of the solution is diazotized with excess standard sodium nitrite solution and hydrochloric acid and the excess sodium nitrite is then back titrated with standard sodium sulphanilate solution.

A-1.1 Reagents

A-1.1.1 *Sodium Hydroxide Solution* — 20 percent (*m/v*).

A-1.1.2 *Standard Sodium Nitrite Solution* — 0.1 N.

A-1.1.3 *Concentrated Hydrochloric Acid*

A-1.1.4 *Standard Sodium Sulphanilate Solution* — 0.1 N.

A-1.1.5 *Starch Iodide Test Papers*

A-1.1.6 *Phenolphthalein Test Papers*

A-1.2 Procedure

Weigh accurately about 22 to 25 g of the sample and dissolve it in water and sodium hydroxide solution until distinctly alkaline to phenolphthalein paper. Transfer the solution to a 1 000-ml volumetric flask and dilute up to the mark with water. Mix well. Pipette 100 ml aliquot of this solution in a 500-ml iodine flask. Then add 150 ml water. Add from a burette sufficient standard sodium nitrite solution so as to provide not more than 2 to 3 ml excess of the solution (to do this, run a direct titration adding hydrochloric acid in the beginning to know the amount of sodium nitrite required and hence excess).

Cool the flask to about 20°C. Quickly add 20 ml concentrated hydrochloric acid. Stopper the flask immediately. Mix well and allow it to stand at 20°C with occasional shaking for a period of 10 min. At the end of this period, test for the presence of sodium nitrite by spotting on starch iodide test paper. Back titrate excess sodium nitrite with standard sodium sulphanilate solution till blue colour on starch iodide test papers just disappears. Add 2 ml excess of sodium sulphanilate and finally back titrate the excess sodium sulphanilate with standard sodium nitrite solution to a distinct blue test on starch iodide test paper that can be obtained repeatedly during a period of 5 min without further addition of sodium nitrite solution. Perform a blank titration in the same manner using the reagents in same proportions under identical conditions.

A-1.3 Calculation

Assay, percent by mass exclusive of α-naphthylamine, calculated on molecular mass 245.2 shall be :

$$(V_1 + V_3 - V_4) \times N_1 - (V_2 \times N_2) \times \frac{245.2}{m}$$

where

V_1 = volume, in ml, sodium nitrite solution added;

V_3 = volume, in ml, sodium nitrite solution added in back titration;

V_4 = volume, in ml, sodium nitrite solution consumed in blank titration;

N_1 = normality of sodium nitrite solution;

V_2 = volume, in ml, sodium sulphanilate solution;

N_2 = normality of sodium sulphanilate solution; and

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

ANNEX B

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

DETERMINATION OF α-NAPHTHYLAMINE

B-0 GENERAL

Two methods are described, one by chemical means another by chromatography.

B-1 DETERMINATION OF α-NAPHTHYLAMINE BY CHEMICAL METHOD

B-1.1 ***Reagents***

B-1.1.1 *Sodium Hydroxide*—20 percent (*m/v*).

B-1.1.2 *Ether Solvent* (*Diethyl Ether*)

B-1.1.3 *Hydrochloric Acid*—20 percent (*m/v*).

B-1.1.4 *Standard Sodium Nitrite Solution*—0.1 N.

B-1.2 Procedure

Weigh 14.3 g of the sample and dissolve in 150 ml of water. Add few drops of sodium hydroxide solution until alkaline to phenolphthalein test paper. Transfer the solution to a separating funnel and extract three times with ether, the first time with 150 ml, the second and the third time with 100 ml each. At the end of each extraction permit the ether to form a clean-cut upper layer. Draw off the lower aqueous layer and wash the combined ether extract with 100 ml of water. Pour the combined ether extract with stirring into 100 ml of hydrochloric acid in a beaker. Boil off the ether by digesting on the water-bath. Cool the solution with crushed ice to 5°C and titrate with standard sodium nitrite solution while stirring mechanically. Add sodium nitrite solution as rapidly as it is consumed. The end point is reached when a drop of the solution spotted on a starch iodide paper produces immediately blue coloured ring which may be repeatedly obtained during 5 min without the further addition of sodium nitrite.

B-1.3 Calculation

α-Naphthylamine content, percent

$$\text{on molecular mass } 143 = \frac{V \times N \times 14.3}{M}$$

where

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

N = normality of standard sodium nitrite solution; and

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

B-2 THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

B-2.1 General

Impurities are determined by thin layer chromatography. Reference may be made to IS 5299 for details of TLC test method to be followed. However, necessary details of test conditions are given below for guidance only:

a)	Product name	:	Naphthionic acid (Sodium salt)
b)	Sample solution (on 100% basis)	:	10% in Acetone + 2% Ammonia (1:1)
c)	Application/volume for spotting	:	10 µl (for sample) 2 µl and 4 µl (for impurities)
d)	Standard	:	Reference standard
e)	Test substance for impurities	:	α–Naphthylamine (0.05% solution in acetone)
f)	Plate type	:	Silica gel G
g)	Eluent	:	Benzene
h)	Elution time	:	30 min
j)	Temperature	:	25 ± 5°C
k)	Detection spray	:	0.01N sulphanilic acid diazo
m)	Evaluation	:	Semi-quantitative
n)	Approximate Rf value:		

1)	Main band	Naphthionic	: Rf	0.0
2)	Impurities	α-naphthylamine	: Rf	0.5

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

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Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. PCD 11 (2055).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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