Doc. No.: PCD 26 (20955) WC
October 2022

BUREAU OF INDIAN STANDARDS

Draft Indian Standard

VINYL SULPHONE ESTER OF 2,5-DIMETHOXY ANILINE — SPECIFICATION

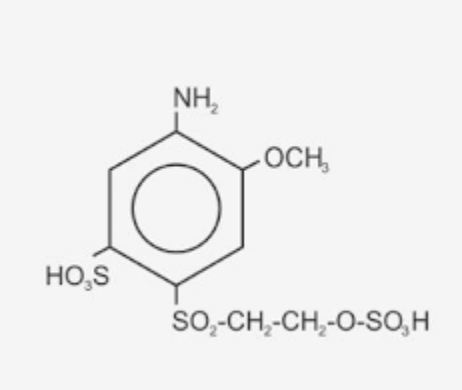
(ICS 71.080.99)

Dye Intermediate Sectional Committee, PCD 26	Last date for Comments **17 December 2022**

FOREWORD

(Formal clauses will be added later)

Vinyl Sulphone ester of 2,5-dimethoxy aniline ($C_{10}H_{15}O_8NS_2$) (DMAVS) is an important intermediate used for making azo dyes. It is also known as 4-amino-2-5-di-methoxyphenyl-*β*-hydroxyethyl sulphone sulphate ester. It is represented by the following structural formula:



Vinyl Sulphone Ester of 2,5-Dimethoxy Aniline
Molecular Mass: 341.36
CAS Number: 26672-24-2

This standard stipulates the requirements and methods of test for vinyl sulphone ester of 2,5-dimethoxy aniline (DMAVS).

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 E, 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

This standard prescribes the requirements, the methods of sampling and testing for vinyl sulphone ester of 2,5-dimethylaniline (DMAVS).

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 : 1992	Reagent grade water — Specification (*third revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)
IS 14887 : 2014	Textiles – High density polyethylene (HDPE) / polypropylene (PP) woven sacks for packing of 50 kg food grains — Specification (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off white to grey powder.

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

Table 1 Requirements for Vinyl Sulphone ester of 2,5-dimethylaniline (DMAVS)
(Clause 3.2, 5.3.1, 5.3.2 *and* 6.2*)*

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Assay (by nitrite value), percent by mass (on dry basis), *Min*	94.0	A
	Or	93.0	B

	Assay (by HPLC[1)]), percent area (on dry basis), *Min*		
ii)	Hydrolysis value, percent by mass (on 100 percent basis), *Min*	90.0	C
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	1.0	D

[1)]In case of disputes, determination of assay by HPLC shall be the referee method.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in HDPE / PP woven sacks (*see* IS 14887). Each bag shall be securely closed.

4.2 Marking

4.2.1 Each bag shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer / supplier, complete address and his recognized trade-mark, if any;
c) Gross, net and tare mass;
d) Lot or batch number;
e) Month and year of manufacturing;
f) Shelf life of the material; and
g) Any other statutory requirement.

4.2.2 All bags in which the material is stored or transported shall also be prominently and clearly marked with red letters ‘DANGER’.

4.2.3 *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

5.2.1 Test for assay by nitrite value shall be conducted on the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, assay by HPLC, hydrolysis value and matter insoluble in sodium carbonate solution 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 nitrite value if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **5.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1**.**

6 TESTS

6.1 PREPARED SAMPLE

Dry the material at (105 ± 1) ^{o}C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

6.2 Tests shall be carried out on the prepared sample (**6.1**) according to the methods prescribed in col 4 of Table 1.

6.3 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)]
DETERMINATION OF VINYL SULPHONE ESTER OF 2,5-DIMETHYLANILINE (DMAVS) CONTENT (ASSAY) BY NITRITE VALUE

A-1 REAGENTS

A-1.1 Concentrated Hydrochloric Acid

A-1.2 Potassium Bromide

A-1.3 Standard Sodium Nitrite Solution, 0.1 N.

A-1.4 Potassium Starch Iodide Indicator Papers

A-1.5 Ice

A-2 PROCEDURE

Weight 10 -14 g dry powder in 250 ml glass beaker. Add distilled water approximately 150 ml and stir with glass rod to make a smooth slurry. Add 20 percent soda ash solution (approximately 7-10 ml) to dissolve the powder to make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 ml of the solution by using pipette in to 1000 ml beaker. Add 200-250 ml distilled water. Add ice cubes to make the temperature around 10°C. Weigh and add 1 g potassium bromide into the cold solution. Add hydrochloric acid to make the *p*H acidic (*p*H around 2-2.5 on *p*H paper) approximately 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the endpoint to put the spot-on starch iodide paper, the end- point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

A-3 CALCULATION

Assay (by nitrite value), percent by mass = $\frac{V_1 \times N_1 \times 341.36}{M \text{ x } 10}$

where

V_1 = volume of standard sodium nitrite solution used in the titration, ml;

N_1= normality of sodium nitrite solution, N; and

M = a mass of the material taken for the test, g

ANNEX B

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

DETERMINATION OF VINYL SULPHONE ESTER OF 2,5-DIMETHYLANILINE (DMAVS) CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-1 OUTLINE OF METHOD

High performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 HPLC, binary gradient liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.1.1 *Column*, C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-2.2 Analytical balance, capable of weighing

B-3 REAGENT

B-3.1 Tetrabutylammonium Hydrogen Sulphate, AR grade.

B-3.2 Acetonitrile, HPLC grade.

B-3.3 Methanol, HPLC grade.

B-3.4 Water, HPLC grade.

B-3.4 Dipotassium Hydrogen Phosphate, AR grade.

B-3.5 Vinyl Sulphone ester of 2,5-dimethylaniline (DMAVS), known purity.

B-4 SAMPLE PREPARATION

Weigh accurately 0.100 g DMAVS sample in 100 ml volumetric flask. Dissolve it in water and make it up to 100 ml with water. Take 2.5 ml of this diluted solution into 100 ml volumetric flask and adjust to 100 ml. The solution so prepared is around 25 ppm.

B-5 BUFFER PREPARATION

Take 0.610 0 g of dipotassium hydrogen phosphate into the beaker. Take 0.2400 g of tetrabutylammonium hydrogen sulphate and add into the beaker. Add water and make it up to 100 ml. Add 70 ml acetonitrile in above solution. Add 7 ml methanol in above solution.

B-6 FLOW RATE, 1.00 ml/min.

B-7 MOBILE PHASE, buffer solution.

B-8 COLUMN OVEN TEMPERATURE, 40^{o}C.

B-9 INJECTION VOLUME, 20 µl.

B-10 RUN TIME, 25-30 min.

B-11 WAVELENGTH, 254 nm (photodiode array detector).

B-12 PEAK TIME

DMAVS : 4.12 min

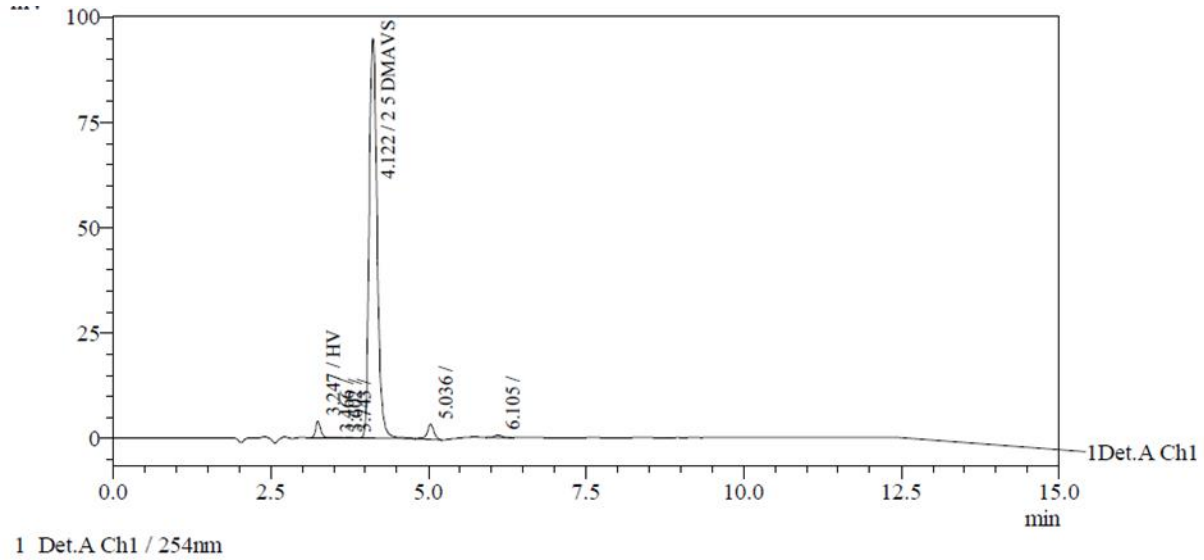


FIG 1 TYPICAL CHROMATOGRAM

B-13 CALCULATION

Calculate the peak area of individual constituent pertaining to DMAVS the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure DMAVS.

$$\text{DMAVS, percent} = \frac{A}{Total\ Area} \times 100$$

where

A = area of DMAVS peak in sample

ANNEX C
[*Table* 1, *Sl No.* (ii)]
DETERMINATION OF HYDROLYSIS VALUE

C-1 REAGENTS

C-1.1 Standard Sodium Hydroxide Solution, 0.1 N

C-1.2. Phenolphthalein Indicator

C-1.3 Ice

C-2 PROCEDURE

Weigh and take 1 g sample in to 1000 ml glass beaker. Add approximately 150-200 ml distilled water into the beaker and dissolve the sample by stirring. Add ice cubes into the beaker to get temperature around 10 ^{o}C. Add phenolphthalein indicator 10-12 drops. Take 0.1 N sodium hydroxide solution into burette. Titrate the sample against 0.1 N sodium hydroxide solution. End point will be pale pink colour. Note down the burette reading. Fill the burette with 0.1 N sodium hydroxide solution and adjust to zero reading. Keep the sample on hot plate and warm the solution to 60^{o}C with constant stirring till the pink colour disappears. Add 0.1 N sodium hydroxide solution from burette slowly under constant stirring, till a stable pink colour is obtained. If necessary, add few drops of phenolphthalein to ascertain the correct end point. Note down this hot burette reading.

A-3.3 Calculation

$$\text{Hydrolysis value, percent by mass} = \frac{V_1 \times N_1 \times 341.36}{M \times 10}$$

where

V_1 = volume of standard sodium hydroxide solution used in the titration (hot), ml;

N_1= normality of sodium hydroxide; and

M = a mass of the material taken for the test, g

ANNEX D
[*Table* 1, *Sl No.* (iii)]
DETERMINATION OF MATTER INSOLUBLES IN SODIUM CARBONATE SOLUTION

D-1 REAGENTS

D-1.1 Sodium Carbonate Solution, 20 percent (*m/v).*

D-2 PROCEDURE

Weigh exactly 10 g of the dry powder sample and pour in to 250 ml beaker. Add 100 ml demineralized water and stir with glass rod to make smooth slurry. Add 20 percent sodium carbonate (soda ash) solution to get *p*H 4.5 to 5.5 and stir well on magnetic stirrer to get clear solution. Add additional demineralized water to make up the volume to 200 ml (approximately). Take whatman filter paper No. 42 of 110 diameter and soak in water and dry in oven till it is dry. Weigh the dried filter paper and note down the weight (initial weight). Place the dried whatman paper in buchner's funnel and add 50 ml water and start the vacuum pump to fix up properly. Pour the dissolved solution from the beaker on the filter paper and suck all the solution. Wash the filter paper by using fresh water from the wash bottle 4-5 times and suck properly. Take out the Whatman filter paper and keep in oven at 105^{o}C for 20 min till it is completely dried. Keep the dried Whatman paper in desiccator and allow to cool to room temperature. Weigh the dried filter paper and note down the weight (final weight).

D-3 CALCULATION

Matter insoluble in sodium carbonate solution, percent by mass = $\frac{m \times 100}{M}$

where

m = mass of the residue, g; and

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

ANNEX E
(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s)	:	
Signal word	:	**DANGER**
Hazard statement(s)	:	H318: Causes serious eye damage H335: May cause respiratory irritation
Precautionary Statement	:	**Prevention:** P261: Avoid breathing dust/fume/gas/mist/vapours/spray P271: Use only outdoors or in a well-ventilated area P280: Wear protective gloves/protective clothing/eye protection/face protection. **Response:** P305 +P351 +P338: If in eyes: Rinse cautiously with water for several minutes. Remove Contact lenses, if present and easy to do. Continue rinsing. P304+P340: If inhaled: Remove victim to fresh air and keep at rest in a position comfortable for breathing **Storage:** P405: Store locked up P403+P233: Store in a well-ventilated place. Keep container tightly closed **Disposal:**

	P501: Dispose of contents/container in accordance with local/regional/national/International regulations. Waste treatment in accordance with national regulations.