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BUREAU OF INDIAN STANDARDS

Draft Indian Standard

SPECIFICATION FOR AMYL SALICYLATE

(Second Revision of IS 3929*)*

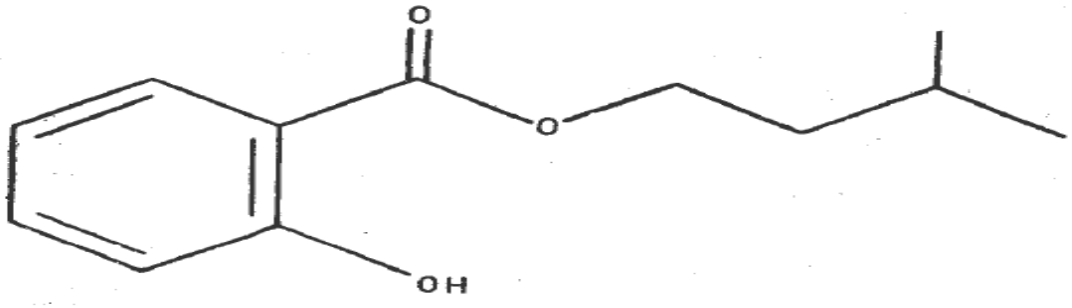
(ICS No. 71.100.60)

Fragrance and Flavour Sectional Committee	Last date for comment is
PCD 18	**21 December 2022**

FOREWORD
(*Formal clauses shall be added later*)

This Indian Standard (second revision) was first published in 1966 and subsequently revised in 1984. In this revision, the gas chromatographic analysis for purity determination of vanillin has been upgraded from Packed Column GC to Capillary Column GC for more accurate results. Also the packing and marking clauses have been updated.

Amy1 salicylate ($C_{12}H_{16}O_3$) is not found in nature. Commercial amyl salicylate chemically is mainly iso-amyl salicylate. It is also known as iso-amyl o-hydroxy-benzoate. It finds extensive application in compounding of floral odours, specially for use in soaps. It has a very It is used in a wide variety of compositions because of its low price and lasting quality. It is represented by the following structural formula.



AMYL SALICYLATE
(Molecular Mass 208·25)

In the preparation of this standard, considerable assistance has been derived from the following publications:

The Givaudan Index, 1978, published by Givaudan – Delawanna Inc., New York.

EOA No. 27-1970 Standard for amyl salicylate (*revised),* published by Essential Oil Association of USA, New York.

Clause **4.3** includes purchaser and seller agreement.

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

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 and the methods of sampling and test for amyl salicylate.

2 REFERENCES

The following standards contain provisions which, through reference in text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
IS 6597 : 2001	Glossary of terms relating to fragrance and flavour industry (Second Revision)
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (First Revision)
IS 326 (Part 7) : 2006/ ISO 1242 : 1999 (Reaffirmed in 2017).	Methods of sampling and test for natural and synthetic perfumery materials Part 7 determination of acid value (Third Revision)
IS 326 (Part 3) : 2006/	Methods of sampling and test for natural and synthetic

ISO 279 : 1998	perfumery materials: Part 3 determination of relative density (Third Revision)
IS 1070 : 1992	Reagent grade water - Specification (Third Revision)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597 : 2001 shall apply.

4 REQUREMENTS

4.1 Description

4.1.1 The material shall be obtained by esterification of iso-amyl alcohol with salicylic acid.

4.1.2 The material shall be a colourless liquid free from sediment, suspended matter and adulterants.

4.2 Solubility

The material shall be clearly soluble in 2 volumes of ethanol (90 percent by volume), when tested as prescribed in IS 326 (Part 6) : 2005.

4.3 The assessment of odour and appearance shall be subject to agreement between the purchaser and seller. The material shall be tested olfactorily, especially for by-odours/ by-notes, and for the presence of adulterants and impurities, if any, as prescribed under **4** and **5** of IS 2284 : 1988.

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

TABLE 1 **REQUIREMENTS FOR AMYL SALICYLATE**
(*Clauses* **4.4** *and* **7.1**)

Sl. No.	**Characterist**	**Requirement**	**Method of test, Ref to**	
			Referred Indian Standard	Annex
(1)	**(2)**	**(3)**	**(4)**	**(5)**
i)	Odour	Agreeable, aromatic odour recalling that of orchids	IS 2284 : 1988	-
ii)	Relative density at 27/27°C [1)]	1.0468 to 1.0518	IS 326 (Part 3) : 2006	-
iii)	Refractive index at 27°C [2)]	1.5033 to 1.5053	IS 326 (Part 5) : 2006	-
iv)	Acid value, *Max*	0.5	IS 326 (Part 7) : 2006	-

v)	Determination of Purity of Amy1 salicylate, percent by mass, *Min*	99	-	A

NOTES —

1. The correction factor for relative density for each degree celcius change in temperature is 0.0006.
2. The correction factor for refractive index for each degree Celsius change in temperature is 0.0004.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in glass-bottles, tins or any other suitable containers as agreed to between the purchaser and the supplier. The material shall not be packed in tin, galvanized iron and mild steel containers.

5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

5.2.1 *Name of the material;*

5.2.2 *Name of the manufacturer and his recognized trade-mark, if any*;

5.2.3 *Batch number and date of manufacture;*

5.2.4 *Net and gross mass of the material;*

5.2.5 *Net volume of the material*; *and*

5.2.6 The containers may also be marked with the Standard Mark.

NOTE **—** The use of the Standard Mark is governed by the provisions of the Bureau of Indian *Standards Act, 2016* and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of Standard Mark may be granted to manufactures or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 Number of Tests

6.2.1 Amyl salicylate content shall be tested on each of the individual samples.

6.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

6.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification if **6.3.1** and **6.3.2** are satisfied.

6.3.1 For ester content, the mean ($\bar{x}$) and range (R) of test results shal1 be calculated:

$$\text{Mean } (\bar{x}) = \frac{Sum\ of\ the\ test\ results}{Number\ of\ the\ test\ results}$$

Range (R) = Difference in the maximum and minimum of the test result.

The lot shall be deemed to have satisfied the requirement for this characteristic if the value of the expression 'X - 0.6 R' is greater than or equal to the minimum limit for amyl salicylate content given in Table 1.

6.3.2 All the test results on the composite sample meet the relevant specification requirements.

7 TEST METHODS

7.1 Tests shall be carried out as prescribed under **4.1, 4.2, 4.3, 4.4** and the appropriate references specified in col 4 and 5 of Table 1.

7.2 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. (***v***)*]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF AMYL SALICYLATE CONTENT

A-1 GENERAL

The chromatographic conditions given here arc for guidance only.

A-2 OUTLINE OF THE METHOD

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and arc detected by suitable means '"hose response is related to the amount of a specific component leaving the column.

A-3 APPARATUS

Gas chromatograph equipped with suitable capillary column and flame ionization detector.

A-4 PROCEDURE

Take 1 µL of sample in a GC Vial and inject into gas chromatograph using following operating conditions.

A-5 GC CONDITIONS FOR NON-POLAR COLUMN

Capillary Column	: Fused silica capillary column coated with non-polar stationary phase (100% Polydimethylsiloxane) SH-RXi-1ms
Column temperature	: -40ºC to 350ºC
Length	: 60 m
Internal diameter	: 0.25 mm
Film Thickness	: 0.25 µm
Injector Temperature	: 240°C
Split Ratio	: 200 : 1
Column flow	: 1 ml/ min
Injection volume	: 0.5 µl
Carrier Gas & Flow	**:** Helium, at constant pressure of 145 kPa
Make up gas flow	: He – 24ml/min, H_2 – 32 ml/min, Zero air – 200ml/min
Column oven Temperature	**:** 60°C to 230°C @ 4ºC/min final hold time 20 min.
Detector type	: FID
Detector Temperature	: 290°C
Run time	: 62.5 min

A-6 GC CONDITIONS FOR POLAR COLUMN

Column	: Fused silica capillary column coated with Polar stationary phase (Poly ethylene glycol) SH- Stabilwax
Column temperature	: -20ºC to 260ºC
Length	: 60 m
Internal diameter	: 0.25 mm
Film Thickness	: 0.25 µm
Injector Temperature	: 240°C
Split Ratio	: 200 : 1
Column flow	: 1.14 ml/ min
Injection volume	: 0.5µl
Carrier Gas & Flow	**:** Helium, at constant pressure of 158 kPa
Make up gas flow	: He – 24ml/min, H_2 – 32 ml/min, Zero air – 200ml/min
Column oven Temperature	**:** 60°C to 230°C @ 4ºC/min final hold time 25 min.
Detector type	: FID
Detector Temperature	: 250°C
Run time	: 67.5 min

NOTE — The above gas chromatographic conditions are suggestive /typical. However, any GC with equivalent column may be used provided standardization/calibration are done after setting up chromatographic conditions for the desired/required resolution.

A-7 CALCULATION

Calculate are percent of each peak by following formulas

Polar and non-polar columns

$$\text{Area \% of individual} = \frac{\textit{Peak area of the indivdual}}{\textit{sum of areas of all the peak in the chromatogram}} \times 100$$

NOTE — The modern instruments are equipped with the software, which automatically calculates area percent of each peak.

CHROMATOGRAMS FOR POLAR AND NON-POLAR COLUMN WOULD BE PROVIDED BY ULTRA INTERNATIONAL LIMITED.