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Draft Indian Standard

COUMARIN — SPECIFICATION

ICS No. 71.100.60

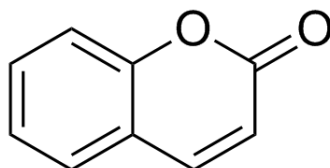
Fragrance and Flavour Sectional Committee
PCD 18

Last date for comments is
24 October 2023

FOREWORD

(Formal Clauses will be added later)

Coumarin (1,2-benzopyrone) is a colorless crystalline solid with a sweet odor resembling the scent of vanilla and a bitter taste. Coumarin is most often used as a fragrance ingredient, where it functions as a fragrance, as a fragrance enhancer, and as a stabilizer. Coumarin is widely used in perfumes, hand soaps, detergents, and lotions at concentrations from 0.01 to 2.4%. It is used to give pleasant aromas to household products or to mask unpleasant odors. Due to its recognizable sharp, sweet flavor, and its possibility to boost vanilla flavor, coumarin has also been used in the production of artificial vanilla extracts.



Coumarin (Molecular Mass 146.14)

Clause 4.1.3 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 draft standard prescribes the requirements and the methods of sampling and tests for Coumarin, used by Fragrance and Flavour Industry.

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.

<i>IS / ISO No.</i>	<i>Title</i>
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
Part 1 : 2022	Sampling (<i>Fourth Revision</i>)
Part 2 : 2023	Preliminary examination of perfumery materials and samples (<i>third revision</i>)
Part 6 : 2005/ ISO 875 : 1999	Evaluation of miscibility in ethanol (<i>Third Revision</i>)
Part 16 : 1989	Determination of melting point and melting range (<i>Second Revision</i>)
1448 (Part 21) : 2019	Methods of test for petroleum and its products (Part 21) Determination of flash point - Pensky - Martens closed cup method (<i>Fourth Revision</i>)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (<i>First Revision</i>)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (<i>Second Revision</i>)

3 TERMINOLOGY

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

4 REQUIREMENTS

4.1 Description

4.1.1 This material shall be free from sediments, suspended matter and adulterants.

4.1.2 The material shall be clear colourless crystalline powder.

4.1.3 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 in IS 2284.

4.2 Solubility — The material (1 gm.) shall be soluble in 15 volumes of ethanol (95 percent by volume), when tested by the method as prescribed in IS 326 (Part 6).

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

Table 1 Requirements for Coumarin
(Clause 4.3)

Sl No.	Characteristic	Requirement	Method of Test Ref. to IS/Annex
(1)	(2)	(3)	(4)
i)	Colour and appearance	White crystalline powder	Visual observation
ii)	Flash Point, °C	160 - 163	IS 1448 (Part 21)
iii)	Melting point, °C	68 - 70	IS 326 (Part 16)
iv)	Determination of Purity of Coumarin, percent by mass, <i>Min</i>	99%	Annex A

5 PACKING AND MARKING

5.1 Packing — The material shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminium, as agreed to between the purchaser and the supplier. The material shall be protected from light and stored in a cool and dry place.

5.2 Marking — Each container so filled shall be clearly marked with the following information:

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

- a) Name of the material
- b) Indication of the source of manufacture
- c) Batch number and date of manufacture
- d) Net and gross mass
- e) Net Volume of the material
- f) BIS Certification Marking

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

6.3 Criteria for Conformity — The lot shall be considered as conforming to the specification if the composite sample satisfies all the requirements specified in this standard.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under **4.1, 4.2**, and the appropriate references specified in col 4 of Table 1.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Clause 4.3, Table 1, Sl. No. (iv)*]

GAS CHROMATOGRAPHIC ANALYSIS OF COUMARIN

A-1 GENERAL

A-1.1 The chromatographic conditions given here are for information and guidance only.

A-1.2 Outline of the Method

A representative port of the material is dissolved in a suitable solvent, if required, and the injected into gas chromatograph equipped with capillary column and flame ionization detector. On completion of chromatographic run, the chromatogram is acquired and area percent of each peak is automatically calculated and presented in the peak table.

A-2 APPARATUS

A-2.1 Gas chromatograph equipped with split / splitless inlet, capillary column and flame ionisation detector.

A-2.2 Gas Chromatographic conditions for Polar column

Column	Capillary, fused silica, length 60 m; internal diameter 0.25 mm, film thickness: 0.25 μ m
Stationary phase	Polar cross bond polyethylene glycol (PEG)
Oven temperature	Initial temperature 60 °C
	Temperature ramping 4 °C/min
	Final temperature 230 °C (hold for 25 min)
Injector temperature	230°C
Split ratio	200: 1
Detector type	Flame Ionisation Detector (FID)
Hydrogen	32 ml/min.
Air	200 ml/min
Make up (He)	24 ml/min
Detector temperature	250°C
Carrier gas	Helium
Carrier gas flow mode	Constant pressure
Carrier gas pressure	158.9 kPa
Injection Volume	0.5 μ l

A-2.3 Gas Chromatographic conditions for Non-Polar column

Column	Capillary, fused silica, length 60 m; internal diameter 0.25 mm, film thickness: 0.25 μ m
Stationary phase	Cross bond 100 % dimethyl polysiloxane
Oven temperature	Initial temperature 60 °C
	Temperature ramping 4 °C/min
	Final temperature 230 °C (hold for 20 min)
Injector temperature	240 °C
Split ratio	200 : 1
Detector type	Flame Ionisation Detector (FID)

Hydrogen	32 ml/min
Air	200 ml/min
Make up (He)	24 ml/min.
Detector temperature	290 °C
Carrier gas	Helium
Carrier gas flow mode	Constant pressure
Carrier gas pressure	145.0 kPa
Injection Volume	0.5 µl

A-3 CALCULATION

Area percent of each peak is automatically calculated and presented in peak table of chromatogram.

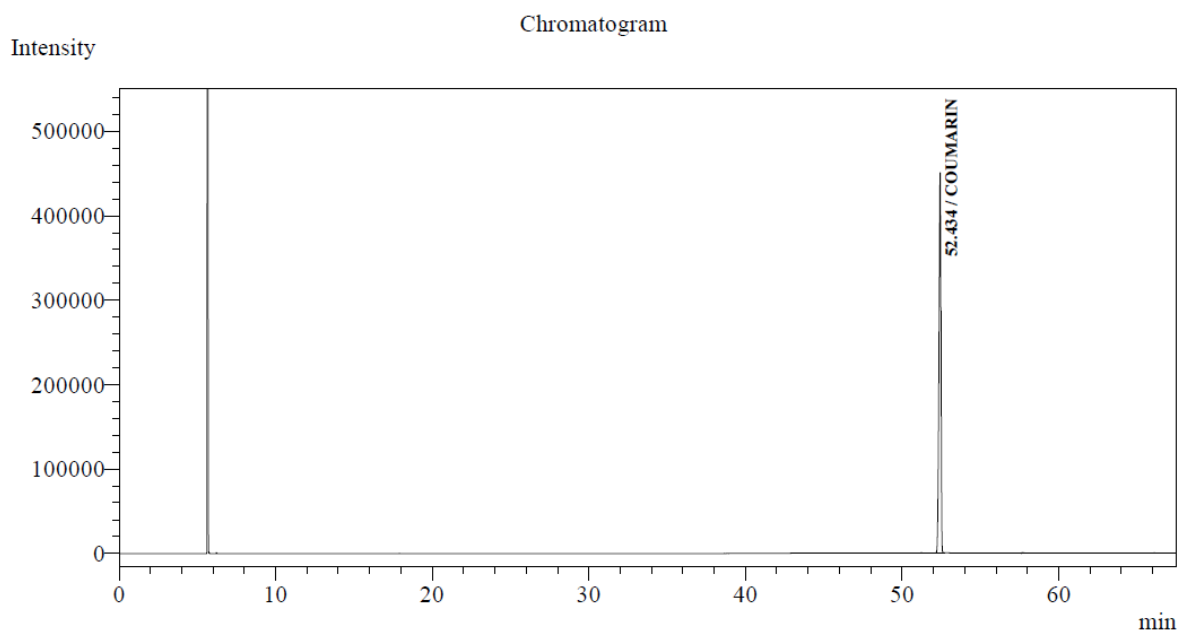


Fig 1 Typical chromatogram acquired on a Polar column

Table 3 Polar Chromatogram Peak Identification

Peak No.	Component
1	Coumarin

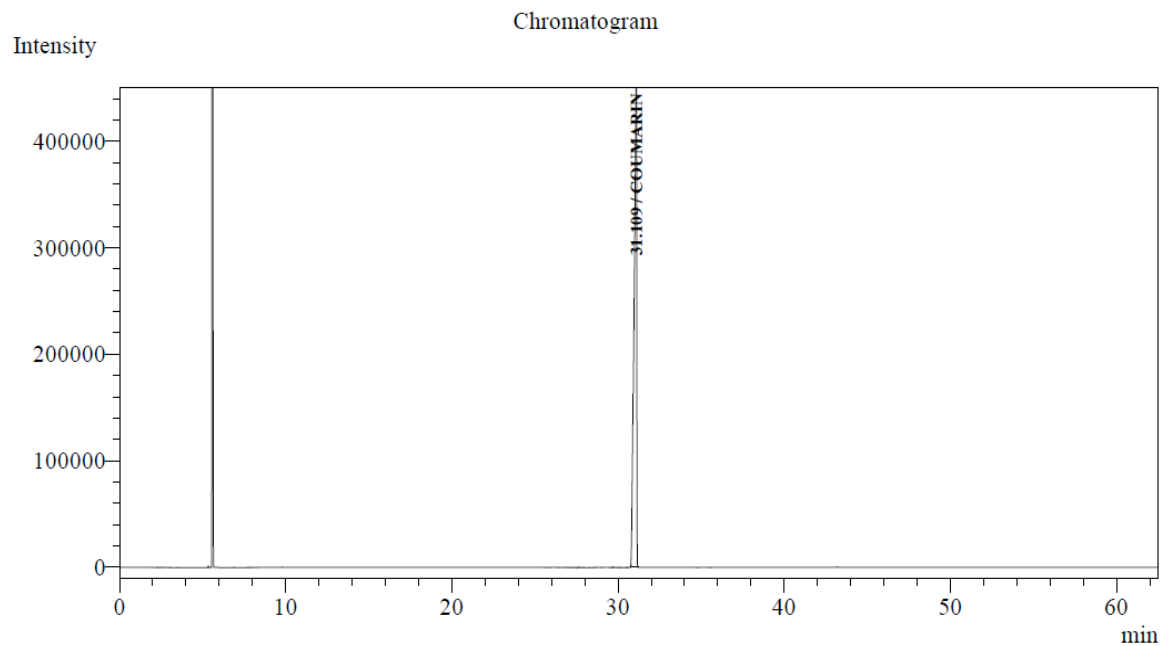


Fig 2 Typical chromatogram acquired on a non-polar column

Table 4 Non- Polar Chromatogram Peak Identification

Peak No.	Component
1	Coumarin