IS 329 (2004): Oil of Sandalwood [PCD 18: Natural and Synthetic Fragrance Materials]

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(तीसरा पुनरीक्षण)

Indian Standard

OIL OF SANDALWOOD — SPECIFICATION

(Third Revision)

ICS 71.100.60

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

September 2004

Price Group 4

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Fragrance Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1952 and first revised in 1961 and subsequent revised in 1993. In the first revision, requirement of ester content was prescribed as 2 percent minimum by mass. However, it was found that users were getting a product with very high ester content, as high as 20 percent. This was probably due to adulteration of oil of sandalwood with other high boiling essential oils of cheaper variety. The presence of these essential oils could not be detected by gas chromatography because of their high boiling range and when estimated with wet methods they were contributing to the total ester content. That is why the ester content determined was showing so high results. To overcome this difficulty the committee has prescribed ester content, calculated as santalyl acetate, 7 percent maximum in this revision. This figure of seven percent is based on the actual value of maximum ester content determined by the genuine manufacturers as well as users over a period of several years.

The Committee felt that steam distillation of oil of sandalwood may help detection of non-volatile adulterants. Therefore, procedure for steam-distillation of material has been covered in this revision as a regular requirement. It was felt that if the oil contains castor oil, glycols or any other heavy adulterant, the same will not distill out whereas distillate in which sandella and diethyl-phthalate come out with oil can be detected by GC for presence of low boiling point adulterants or extraneous materials.

Further, it was noted that maximum alcohol content in natural sandalwood oil is 94 percent on an average 90 to 92 percent alcohol is present in the oil. In case, the oil of sandalwood shows presence of more than 94 percent alcohol, the material must be checked for unwanted additives.

A procedure for detection of adulteration in oil of sandalwood with glycols as developed by AGMARK is given in Annex A for guidance.

In this revision, other requirements of oil of sandalwood have also been updated in order to bring it at par with the current oil of sandalwood available in the market. GC method of analysis has been incorporated in this revision.

Sandalwood is widely grown in South India mainly in Karnataka and Tamil Nadu. The tree grows to a height of 30 feet has a girth of one metre, takes 60 years to grow and gives out fragrance for over 80 years. Of the current total world production of approximately 100 metric tonne of sandalwood oil 40 metric tonne is produced in India. Amongst the oils exported, sandalwood still holds the most enviable position as the queen of essential oils and is virtually monopoly of India.

Indian oil of sandalwood is the product of *Santalum album Linn.* This specific botanical origin and the limits of physicochemical characteristics of the oil distinguish it for its much prized characteristics and intense odour. While preparing the third revision of this standard ISO 3518 : 2002 'Oil of Sandalwood (*Santalum album Linnaeus*)' has been consulted. This standard differs from ISO 3518 mainly in respect of the following:

a) The temperature for determination of relative density and refractive index has been indicated as 27°C against 20°C in ISO standard. Accordingly the requirements for these properties have also been changed.

b) No temperature indication has been specified for the determination of optical rotation whereas ISO standard specifies the determination of this property is at 20°C. Accordingly, the range for optical rotation has also been changed.

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 values in the standard.

Indian Standard

OIL OF SANDALWOOD — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for oil of sandalwood.

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 revisions 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*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Determination of relative density (*second revision*)
(Part 4) : 1980	Determination of optical rotation (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility in ethanol (*second revision*)
(Part 8) : 1980	Determination of ester value, content of esters and combined alcohols (*second revision*)
(Part 9) : 1980	Determination of ester value after acetylation and free alcohols (*second revision*)
(Part 19) : 1998	Gas chromatographic analysis of perfumery materials
1070 : 1992	Reagent grade water — Specification (*third revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

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

4 REQUIREMENTS

4.1 Description

4.1.1 The oil of sandalwood shall be obtained by steam distillation of the ground heartwood and roots of *Santalum album* Linn., fam. *santalaceae*.

4.1.2 The oil of sandalwood shall be clear liquid free from sediment, suspended matter, separated water and added adulterants, when tested as per IS 326 (Part 2).

4.2 Solubility

The material shall be soluble in 5 volumes of ethyl alcohol (70 percent by volume) at 27 °C when tested as prescribed in IS 326 (Part 6).

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

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be supplied in air-tight containers, preferably glass, tin-lined or aluminum, permitting a minimum of air space, as agreed to between the purchaser and the supplier.

5.1.2 Direct contact of the material with the galvanized or mild steel containers shall be avoided.

5.1.3 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

The container shall be marked with the following information:

a) Name of the material;
b) Manufacturer's name and address and/or his recognized trade-mark, if any;
c) Net mass of the material;
d) Month and year of manufacture;
e) Batch number;

f) Percentage of free alcohol, calculated as santalol, and of ester, calculated as santalyl acetate; and

g) Cautionary notice if any.

5.2.1 *BIS Certification Marking*

The sheets may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the rules and regulations made thereunder. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturer or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the materials, each sample containing not less than 50 ml shall be drawn as prescribed under IS 326 (Part 1).

7 NUMBER OF TESTS

7.1 Test for the determination of all the characteristics shall be conducted on the composite sample as prescribed in 4.1, 4.2 and Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be used in tests (*see* IS 1070).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of the analysis.

Table 1 Requirements for Oil of Sandalwood

(*Clauses* 4.3 *and* 7.1)

Sl No.	Characteristics	Requirements	Method of Test, Ref to	
			Indian Standard	Annex
(1)	(2)	(3)	(4)	(5)
i)	Colour and appearance	Nearly colourless to golden yellow, somewhat viscous oily liquid	326 (Part 2)	—
ii)	Odour	Pleasant, sweet, woody and persistent	2284	—
iii)	Relative density at 27 °C/27 °C[1)]	0.963 5 – 0.977 5	326 (Part 3)	—
iv)	Optical Rotation	–20 ° to –15 °	326 (Part 4)	—
v)	Refractive Index at 27 °C[1)]	1.500 0 – 1.507 0	326 (Part 5)	—
vi)	Esters, calculated as santalyl acetate ($C_{17}H_6O_2$), percent by mass, *Max*	7	326 (Part 8)	—
vii)	Total alcohol, calculated as santalols ($C_{15}H_{24}O$) percent by mass, *Min*	90	326 (Part 9)	—
viii)	Steam-distillation residue for 10 h, percent by mass, *Max*	5	—	B
ix)	Major chemical constituents by GLC analysis:			
	1) cis – α-Santalol, percent	41 – 55	—	C
	2) cis – β-Santalol, percent	16 – 24	—	C

NOTES

1 The correction factor for relative density [*see* IS 326 (Part 3)] and refractive index [*see* IS 326 (Part 5)] for each degree Celsius change in temperature is 0.000 64 and 0.000 38 respectively.

2 2 g of anhydrous sodium acetate shall be used for acetylation and 1.2 g of acetylated oil be taken for determining the saponification value after acetylation.

ANNEX A

(Foreword)

DETECTION OF ADULTERANTS IN OIL OF SANDALWOOD

A-1 GENERAL

Thin layer chromatographic (TLC) method has been successfully used for detecting various adulterants in essential oils. According to this procedure, genuine samples of oil of sandalwood may be taken as control samples and various possible glycol adulterants may be mixed in varying concentrations to prepare the known adulterated samples (generally polyethylene glycol 400 and polyethylene glycol 600 are used as adulterants). TLC pattern of the samples to be tested are compared with the TLC pattern of known adulterated samples. The comparison indicates the concentration of glycol adulterant present, if any.

A-2 OUTLINE OF THE METHOD

Adulteration of polyethylene glycol up to 1 percent can be detected by this method. A solution of known concentration of sandalwood oil and polyethylene glycols shall be prepared in absolute alcohol. The above prepared solutions shall then be spotted on TLC plates. The plates shall then be developed in a TLC tank containing a suitable developing solvent mixture. The objective is to separate the polyethylene glycols completely from the constituents of natural sandalwood oil. Solvent mixture of benzene, ethyl alcohol and *n*-butanol shall be used. The TLC plates may then be developed to a height of a 12 cm from the point of application. Then the plates shall be taken out from the TLC tank, air dried and sprayed with vanillin-sulphuric acid solution. The plates shall then be heated for 5 min in an air oven for development of the spots. It may be observed that all the constituents of natural sandalwood oil get migrated to the solvent front. The plates may then be cooled to room temperature and then sprayed with iodine-sulphuric acid solution. Polyethylene glycols form a dark brown spot at the place of spotting. These compounds do not migrate at all and at higher concentration are found to form a streak from the point of application. Genuine sandalwood oil leaves no spot at this point.

A-3 APPARATUS AND REAGENTS

A-3.1 TLC Plates, glass plates, 10 × 20 cm coated with slurry of silica gel G to a thickness of 250 µm, allowed to set at room temperature for 30 min, activated at 105 °C in an air oven for one hour and desiccated.

A-3.2 Developing Tank, Micro Pipettes, Spray Bottles, etc

A-3.3 Developing Solvent Mixture, mix benzene, ethyl alcohol and *n*-butanol in the ratio 50 : 12 : 5.

A-3.4 Spray Reagent

A-3.4.1 *Vanillin-Sulphuric Acid Solution*

1.0 g of vanillin is dissolved in 100 ml of absolute alcohol to which 2 ml of concentrated sulphuric acid has been added.

A-3.4.2 *Iodine-Sulphuric Acid Solution*

Mix equal volume of 0.1 N iodine solution and 10 percent sulphuric acid.

A-4 PROCEDURE

A-4.1 Prepare a 10 percent (*v/v*) solution of suspected sandalwood oil in absolute alcohol. Five µl of this solution is spotted on the TLC plate and the plate is then developed to a height of 12 cm from the point of application in a chromatographic tank containing the developing solvent mixture (*see* **A-3.3**). For better chamber saturation, the tank may be lined with filter paper soaked in the developing solvent mixture. The plate is removed from the tank, air dried and then sprayed with spray reagent vanillin-sulphuric acid solution (*see* **A-3.4.1**). It is then placed in an air oven at 105 °C for 5 min to develop the spots. The plate is removed from the oven, air cooled to room temperature and then sprayed with spray reagent iodine-sulphuric acid solution (*see* **A-3.4.2**). Polyethylene glycol 400 and polyethylene glycol 600 if present in the oil either singly or in combination will give a dark brown spot at the point of application. Under the experimental conditions, the constituents of genuine sandalwood oil will move along with the solvent front leaving the adulterants at the spot front.

This method is simple, less time consuming, specific and can be used for routine analysis.

A-4.2 This experiment may be repeated with 5, 2 and 1 percent adulteration of polyethylene glycols in sandalwood oil. Genuine oil and pure adulterants may also be spotted as control. In all the cases dark brown spot due to polyglycols would be clearly visible. Adulteration below 1 percent is difficult to detect by this procedure.

ANNEX B

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

DETERMINATION OF STEAM-DISTILLATION RESIDUE

B-1 OUTLINE OF THE METHOD

The steam-distillation of oil of sandalwood may help detection of non-volatile adulterants, if any. In case the oil contains castor oil, glycols or any other heavy adulterant, the same will not distill out whereas distillate in which sandella and diethylphthalte come out with oil can be detected by GC for presence of low boiling point adulterants or extraneous materials.

B-2 APPARATUS (*see* Fig. 1)

The parts and dimensions are as follows:

B-2.1 Distillation Flask

Spherical, of 1 000 ml capacity with ground neck, taper or ground socket 1 in 10, internal diameter of larger end 33.45 to 34.65 mm.

B-2.2 Still Head, Condenser, Graduated Measuring Tube and Return-Flow Tube in One Piece

a) *Ground joint, A* – accurately fitting in ground neck of flask taper 1 in 10; external diameter of smaller end 31.0 to 31.2 mm and minimum length of ground zone 34 mm.

b) *Tube, AC* – length 190 to 210 mm and internal diameter 13 to 15 mm.

c) *Tube, CED* – length 145 to 155 mm internal diameter 7 to 8 mm.

d) *Condenser, FG* – length 145 to 155 mm, internal diameter 14 to 15 mm and internal diameter at constrictions 9 to 10 mm.

e) *Stopper, K' and Tubular, K* – taper 1 in 10, nominal internal diameter of larger end of ground socket 7.5 mm, nominal external diameter of small end of ground cone 5.7 mm and angle GHK 30° to 40°.

f) *Tube, GH* – internal diameter 7 to 8 mm.

g) *Measuring Tube, JL* – length of graduated portion 145 to 15 mm. Capacity 4 ml, sub-divided into 0.05 ml.

h) *Tube, HJ* – length 30 to 40 mm.

j) *Tube, BL* – internal diameter 7 to 8 mm, vertical height of junction *B* below top graduation mark of 20 to 25 mm, and distance between vertical axes 100 to 110 mm.

B-2.3 Burner

A suitable luminous burner with chimney and sensitive regulating tap.

B-2.4 Stand

A retort stand with asbestos covered ring and clamp carrying a piece of metal tubing *N* connected by a short length of rubber tubing with water inlet tube of the condenser jacket.

B-3 PROCEDURE

Sample of the essential oil bearing material sufficient if possible to yield 1 to 4 ml of volatile oil. To this add water to fill the flask slightly less than half full. Small amount (usually 1 ml) of xylene is added in case of oils with components heavier than water. Connect the flask to the still-head, remove stopper *K'*, and run water into the tube until it once flows at *B*. Heat the flask with frequent agitation, until ebullition commence and continue the distillation at the rate of 65 to 75 drops per minute. Rotate the flask occasionally to wash down any material adhered to the upper part of walls. Distill off for not less than 4 h and at the end of the period discontinue heating and after at least 5 min, read of the volume of oil in the graduated portion of the tube. Continue the distillation until two consecutive readings taken at an hour interval show no change in the volume of the oil collected in the graduated portion of the tube. Cool to room temperature and allow to stand until oily layer is clean. Measure the volume of the oil estimated to the nearest 0.02 ml.

NOTE — If separation of oil and water is not satisfactory, stir the liquid in the graduated tube with wire insertion through the condenser. If necessary add a few millilitres of saturated sodium chloride solution. If foaming occurs, the use of a suitable non-volatile anti-foaming agent is recommended.

B-4 CALCULATION

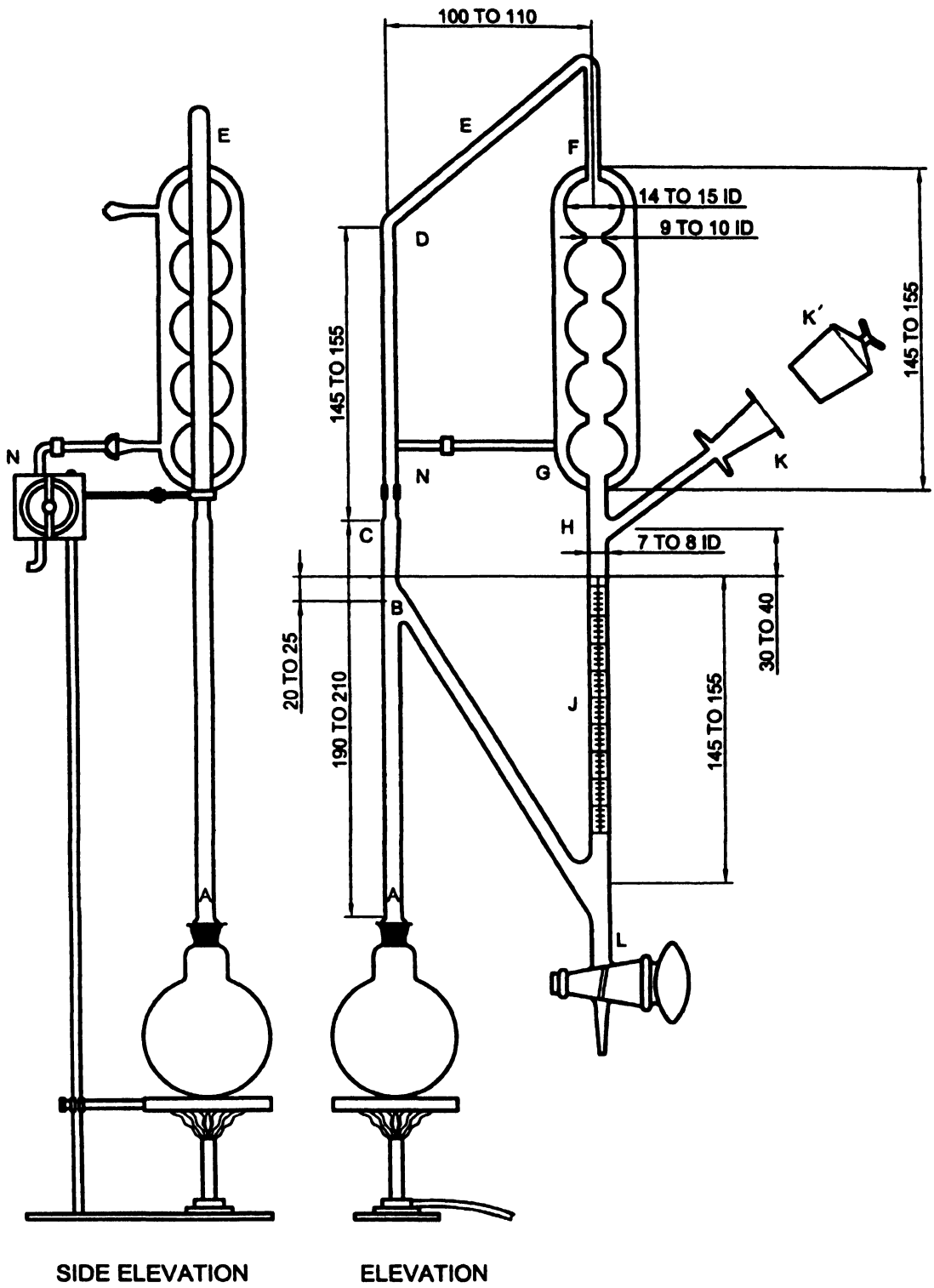
B-4.1 Essential oil content, percent, $(v/m) = \frac{a \times 100}{b}$

where

a = volume, in ml, of steam volatile oil collected through steam-distillation; and

b = mass, in g, of the sample taken.

B-4.2 Steam-distillation residue, percent $= 100 - \frac{a \times 100}{b}$



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF ESSENTIAL OIL CONTENT AND STEAM-DISTILLATION RESIDUE

ANNEX C

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

GAS CHROMATOGRAPHIC ANALYSIS OF OIL OF SANDALWOOD

C-1 GENERAL

The chromatographic analysis is given in capillary column. The chromatographic conditions given here are for information and guidance.

C-2 PROCEDURE

C-2.1 The analysis shall be done as per IS 326 (Part 19). The typical chromatograms for oil of sandalwood on capillary column (polar and non-polar column) with the following chromatographic conditions is shown in Fig. 2 and Fig. 3.

C-2.1.1 *Gas Chromatographic Conditions on Capillary Column*

Column	: Carbowax-20M,length 25m, internal diameter 0.2 mm, film thickness 0.2 μm
Stationary phase	: Polyethylene glycol
Carrier gas	: Nitrogen
Column temperature (programme with 2 °C/min temperature rate)	: 80 °C – 200 °C
Injection port temperature	: 210 °C
Detector used	: Flame ionization detector
Detector temperature	: 220 °C

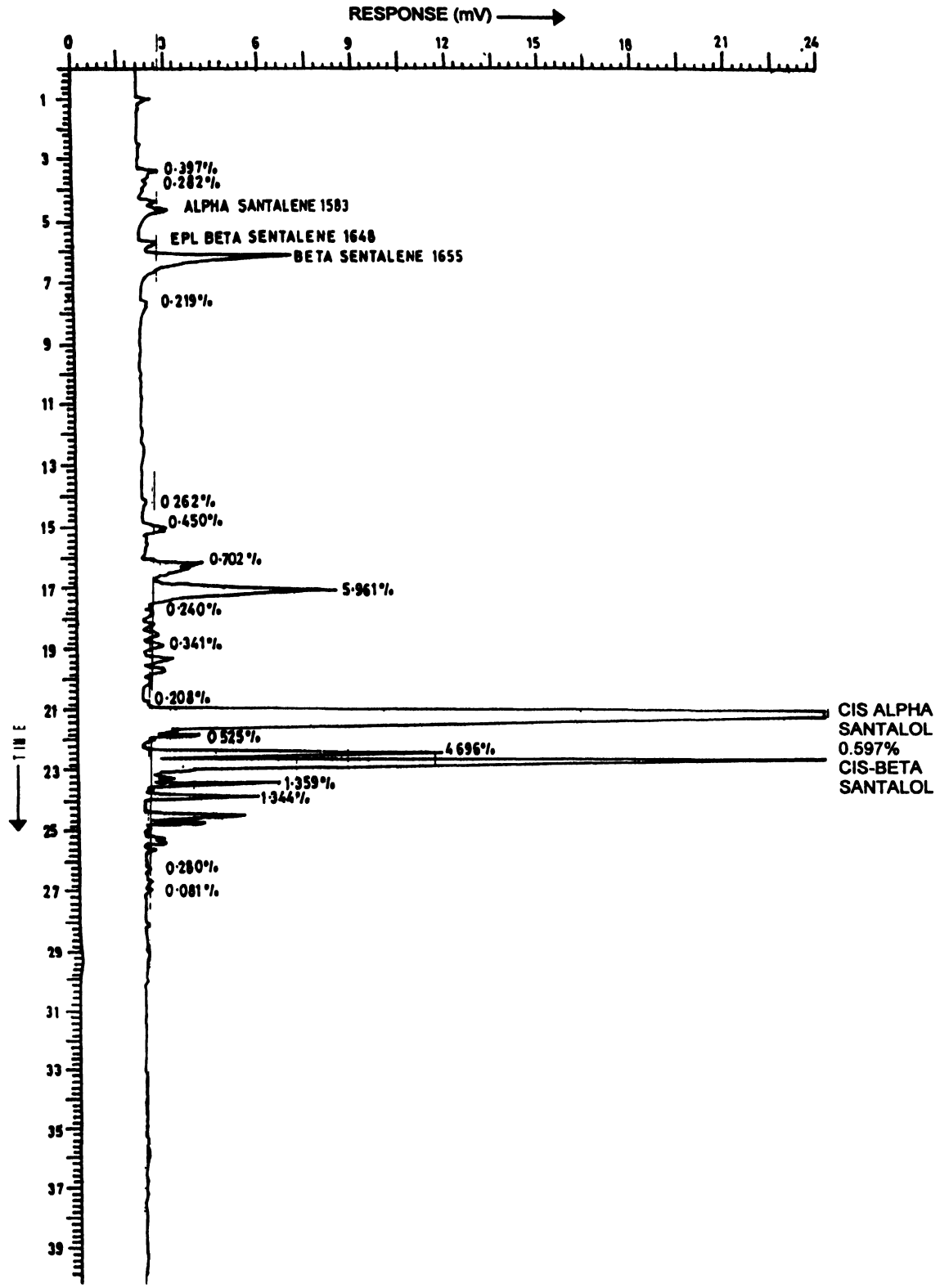


FIG. 2 OIL OF SANDALWOOD — GC PROFILE ON POLAR COLUMN

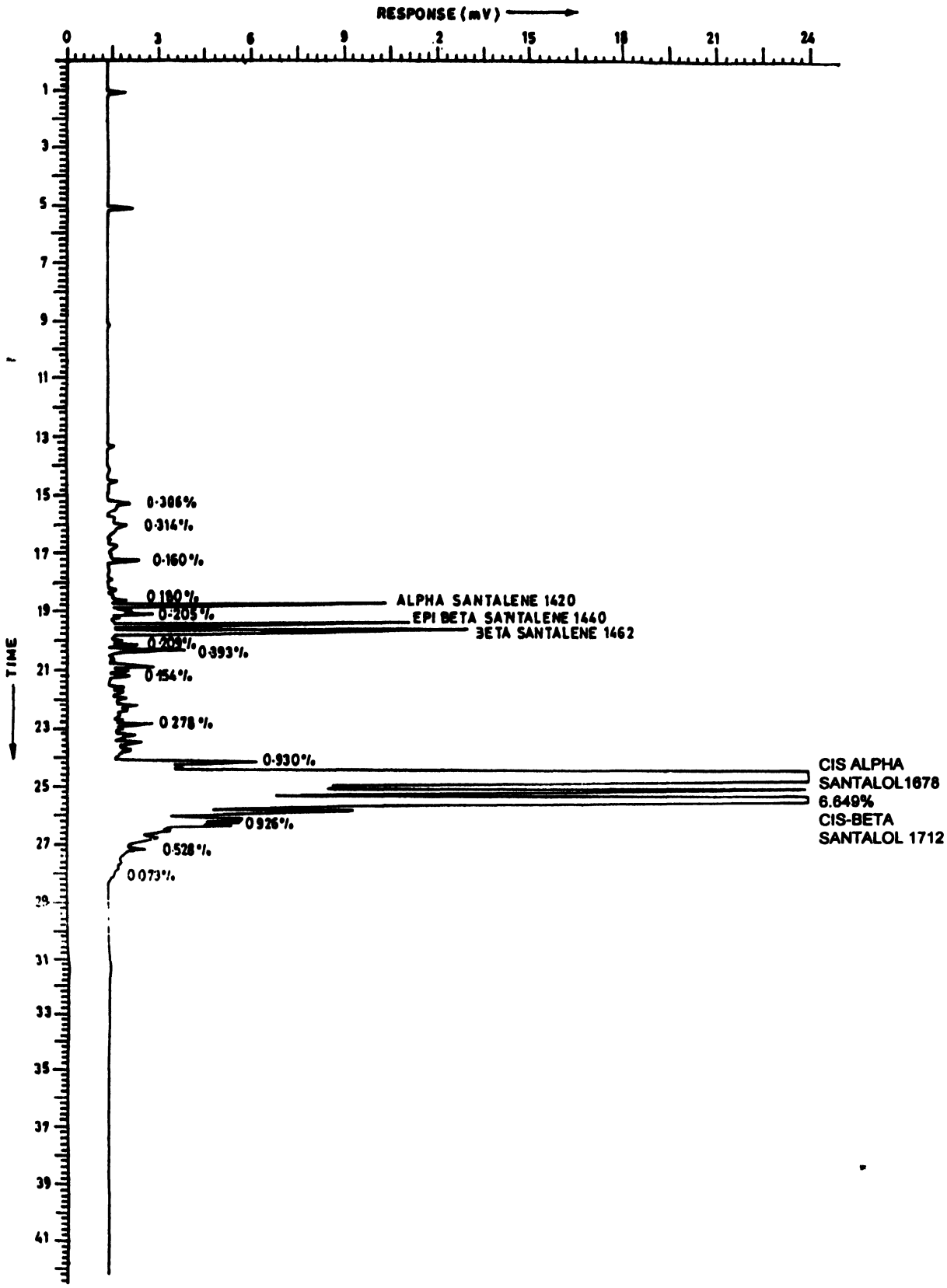


FIG. 3 OIL OF SANDALWOOD — GC PROFILE ON NON-POLAR COLUMN

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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 18 (2018).**

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *website* : www.bis.org.in

Regional Offices:		Telephones
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617, 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160022	260 3843, 260 9285
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Printed at Simco Printing Press, Delhi