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Draft Indian Standard

OIL OF CITRONELLA (JAVA) — SPECIFICATION

(Second Revision of IS 512)

(ICS No. 71.100.60)

Fragrance and Flavour Sectional Committee, PCD18	Last date for Comments: **24 October 2023**

FOREWORD

(*Formal clauses will be added later*)

Oil of citronella (Java) is one of the most important essential oils used in perfumery industry. It acts as main starting material for the production of numerous important perfumery materials such as geraniol, citronellal and their derivatives. The oil as such also finds extensive use in perfumery, various household cleaners, technical products, insecticides, etc.

Earlier this oil was imported in India. However, in the recent past, citronella grass of Java type is being cultivated extensively in various parts of India particularly in Assam (Eastern regions). The oil produced in India has been found to be, by and large, comparable with the imported Java type oil.

This standard was first published in1954 and subsequently revised in 1961 after incorporating the new requirement for steam distillation residue. The revised standard covered two types of citronella oil namely, Ceylon type and Java type as the oil produced in Sri Lanka approximates closely to Java type oil. During the second revision, the Committee decided to confine the requirements to only Java type oil of citronella and eliminating the specification of Ceylon type oil of citronella since the latter is not yet produced commercially in India.

In this (third) revision, the gas chromatographic method has been upgraded from Packed Column GC to Capillary Column GC for more accurate results, which are being progressively used in the country. Besides, the values for Optical Rotation, Refractive Index and Relative Density have been reported at 20°C in addition to 27°C in order to facilitate international trade, since the fragrance and flavor ingredients are traded at international level quoting the Optical Rotation, Relative Density and Refractive Index at 20ºC rather than at 27ºC as prescribed in the Indian standards. The requirement for Flash Point has also been incorporated in this revision.

In the preparation of this standard, considerable assistance has been derived from E.O.A. No. 14, Citronella Oil Java type', published by Essential Oil Association of USA, New York and ISO 3848: 2016 Oil of Java citronella, published by the International Organization for Standardization.

The composition of the Committee, responsible for the formulation of this standard is given at Annex C (will be added later).

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 method of sampling and test for oil of citronella (Java).

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All 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 standard indicated below:

IS No.	***Title***
IS 326 (Part 1) : 2022	Methods of sampling and test for natural and synthetic perfumery materials Part 1 sampling (*Fourth Revision*)
IS 326 (Part 2) : 2023	Methods of sampling and test for natural and synthetic perfumery materials Part 2 Preliminary Examination of Perfumery Materials and Samples (*Third Revision*)
IS 326 (Part 3) : 2006 / ISO 279 : 1998	Methods of sampling and test for natural and synthetic perfumery materials: Part 3 determination of relative density (*Third Revision*)
IS 326 (Part 4) : 2005 / ISO 592 : 1998	Methods of sampling and test for natural and synthetic perfumery materials: Part 4 determination of optical rotation (*Third Revision*)
IS 326 (Part 5) : 2006 / ISO 280 : 1998	Methods of sampling and test for natural and synthetic perfumery materials: Part 5 determination of refractive index (*Third Revision*)
IS 326 (Part 6) : 2005 / 875 : 1999	Methods of sampling and test for natural and synthetic perfumery materials: Part 6 evaluation of miscibility in ethanol (*Third Revision*)
IS 326 (Part 9/Sec 1) : 2017 / 1241 : 1996	Methods of sampling and test for natural and synthetic perfumery materials: Part 9 determination of ester value and free alcohols Section 1 determination of ester values, before and after acetylation, and evaluation of the contents of free and total alcohols (*Third Revision*)

IS 326 (Part 9/Sec 2) : 2017 / ISO 3794 : 1976	Methods of sampling and test for natural and synthetic perfumery materials: Part 9 determination of ester value and free alcohols Section 2 estimation of free alcohols content by determination of ester value after acetylation (*Third Revision*)
IS 326 (Part 11/Sec 1) : 2017 / ISO 1271 : 1983	Methods of sampling and test for natural and synthetic perfumery materials: Part 11 determination of carbonyl value Section 1 free hydroxylamine method (*Third Revision*)
IS 326 (Part 11/Sec 2) : 2017 / ISO 1279 : 1996	Methods of sampling and test for natural and synthetic perfumery materials: Part 11 determination of carbonyl value Section 2 potentiometric methods using hydroxylammonium chloride (*Third Revision*)
IS 326 (Part 15) : 1984	Methods of sampling and test for natural and synthetic perfumery materials: Part 15 detection of petroleum and mineral oils (*Second Revision*)
IS 326 (Part 26) : 2017	Methods of sampling and test for natural and synthetic perfumery material: Part 26 general guidance on determination of flashpoint
IS 6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*Second Revision*)
IS 1070 : 2023	Reagent Grade Water Specification (*Fourth Revision*)
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*First Revision*)

3 TERMINOLOGY

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

4 REQUIREMENTS

4.1 Description

4.1.1 The oil shall be obtained by hydro-steam distillation of the freshly out orpartially dried citronella grass of Java type ofbotanical species –*Cymbopogon winterianus*Jowitt (syn. *Andropogon nardus* auct. in the part non Linn.), Var. – mahapengiri.

4.1.2 The oil shall be a clear, sometimes slightly opalescent liquid, free from sediment, suspended matter, separated water and adulterants, when examined as prescribed in IS 326 (Part. 2).

4.1.3 The assessment of odour and appearance shall be subject to agreement between the purchaser and seller. The oil shall be tested olfactorily, especially for by-notes, and for the presence of adulterants and impurities, if any, as per IS 2284.

4.2 Solubility– The oil shall be soluble in1 to 2 volumes of ethanol (80 percent by volume) when tested as prescribed in IS 326 (Part 6).

4.3 The oil shall comply with the requirementsgiven in Table 1when tested according to the methods given in col **4** and **5** of Table 1.

TABLE 1 REQUIREMENTS FOR OIL OF CITRONELLA (JAVA)
(*Clause* 4.3)

Sl. No.	**CHARACTERISTICS**	**REQUIREMENT**	**METHOD OF TEST, REF TO**	
			Indian Standard	**Annex**
(1)	(2)	(3)	(4)	(5)
i)	Colour and appearance	Pale yellow to light tan clear liquid	Visual Observation	–
ii)	Odour	Characteristic citrus grass with rose undertone	IS 2284 :1988	–
iii)	Relative density (1)		IS 326 (Part 3) :2006	–
	at 20 ºC	0.8852 to 0.8961		
	at 27 ºC	0.8801 to 0.8908		
iv)	Optical rotational		IS 326 (Part 4): 2005	–
	at 20 ºC	–5° to +1º		
	at 27 ºC	– 0·5 to – 5º		
v)	Refractive index(2)		IS 326 (Part 5) : 2006	
	at 20 ºC	1.46936 to 1.47410		
	at 27 ºC	1.47027 to 1.47573		
vi)	Total acetylizable matter calculated a geraniol, percent by mass, *Min*	85	IS 326 (Part 9/Sec 1) : 2017 IS 326 (Part 9/Sec 2) : 2017	–
vii)	Total carbonyl compounds calculated as citronellal percent by mass,*Min*(using 1 g of test sample, standing time of 15 minutes, by hydroxylammonium chloride method)[3]	35 to 45	IS 326 (Part 11/Sec 1) : 2017 IS 326 (Part 11/Sec 2) : 2017	–
viii)	Mineral oil	No readable separation of mineral oil	IS 326 (Part 15) : 1984	–
ix)	Steam distillation residue percent by mass, *Max*	3	–	B

x)	Flash Point	(+) 84.5 ºC	IS 326 (Part 26) : 2017	–

NOTES

1. The correction factor for relative density for each degree Celsius change in temperature is 0.00062.

2. The correction factor for refractive index for each degree Celsius change in temperature is 0.00044.

3.Low values are obtained for this isolate, if the usual hydroxylamine hydrochloride technique is used. Fairly satisfactory results may be obtained, if the solution is well cooled and the titration is carried out at low temperature (like –10°C).

4.4Chromatographic Profile

Carry out the Gas Chromatography analysis of the essential oil as per Annex A. The proportions of the components in the chromatogram are given in Table 2 and Table 3. The Marker chemicals identified in Oil of Citronella (Java) are listed as per their order of elution in a non-polar phase capillary column coated with poly dimethyl siloxane as stationary phase and a polar phase capillary column coated with polyethylene glycolas stationary phase respectively.

5 PACKING AND MARKING

5.1 Packing – The material hall be supplied in glass bottles, or in suitable containers as agreedto between the purchaser and the supplier. However,aluminium container shall be avoided.The containers shall be tightly closed and nearlyfull.The material shall also be well-protected from light and stored in a cool place.

5.2 Marking –Each container so filled shallbear legibly and indelibly the following information:

a) *Name of the material;*
b) *Name of the manufacturer and his recognized trade-mark;*
c) *Net and gross mass of the material;*
d) *Net volume of the material;*
e) *Batch no.;*
f) *Manufacturing date.*
g) *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 license 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 RepresentativesamplesofthematerialshallbedrawnasprescribedinIS326(Part1).

6.2 Number of Tests

Tests for the determination of all the characteristics shall be conducted on the composite sample as prescribed in 4.3 and Table 1.

8 TEST METHODS

8.1 Tests shall be conducted as prescribed under **4.1.2**, **4.1.3**, **4.2**, **4.3**, and **4.4** and the appropriate references to relevant parts and Annexes of the standards as given in co1 **4** and **5** of Table 1.

8.2 Quality of Reagents– Unless specified otherwise, pure chemicals and distilled water *(see*IS1070) 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*4.4)
CAPILLARY GAS CHROMATOGRAPHIC ANALYSIS OF OIL OF LEMONGRASS

A-1 GENERAL

The chromatographic conditions given here are 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 1µL of sample in a GC vial and inject into gas chromatograph using following operating conditions.

A-5 GC CONDITIONS FOR NON-POLAR CAPILLARY COLUMN

Capillary Column	: Fused silica capillary column coated with non-polar stationary phase (5% diphenyl)
Column length	: 30 m
Internal diameter	: 0.25 mm
Film Thickness	: 0.25 µm
Injector Temperature	: 240°C
Split Ratio	: 40:1
Injection volume	: 0.5µl prepared in suitable solvent
Carrier Gas & Flow	:Hydrogen/ nitrogen at 1 ml/min constant flow
Make up gas flow	: Nitrogen at 29 ml/min
Column oven Temperature	:60°C to 230°C @ 3ºC/min final hold time 2 min.
Detector type	: FID (Flame Ionization)
Detector Temperature	: 290°C

A-6 GCCONDITIONSFORPOLARCAPILLARYCOLUMN

Column	: Fused silica capillary column coated with Polar stationary phase (Polyethyleneglycol)
Column length	: 60 m
Internal diameter	:0.25 mm
Film Thickness	: 0.32 µm
Injector Temperature	:250°C
SplitRatio	:40:1
Injectionvolume	:0.5µlin a suitable solvent
CarrierGas&Flow	**:**Hydrogen/ nitrogen at 1 ml/min constant flow
Make upgasflow	: Nitrogen at 29 ml/min
ColumnovenTemperature	**:**40°C to 120 (9min) at 3ºC/min, 120°C to 140°C (2min) at 2ºC/min, 140°C to 250°C @5ºC/min final hold time 2 min.
Detectortype	:FID (Flame Ionization)
DetectorTemperature	: 250°C

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

A-7 CALCULATION:

Calculatearea percent of each peak by following formula.

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

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

FIG. 1 – TYPICAL CHROMATOGRAM ACQUIRED ON A NON -POLAR COLUMN

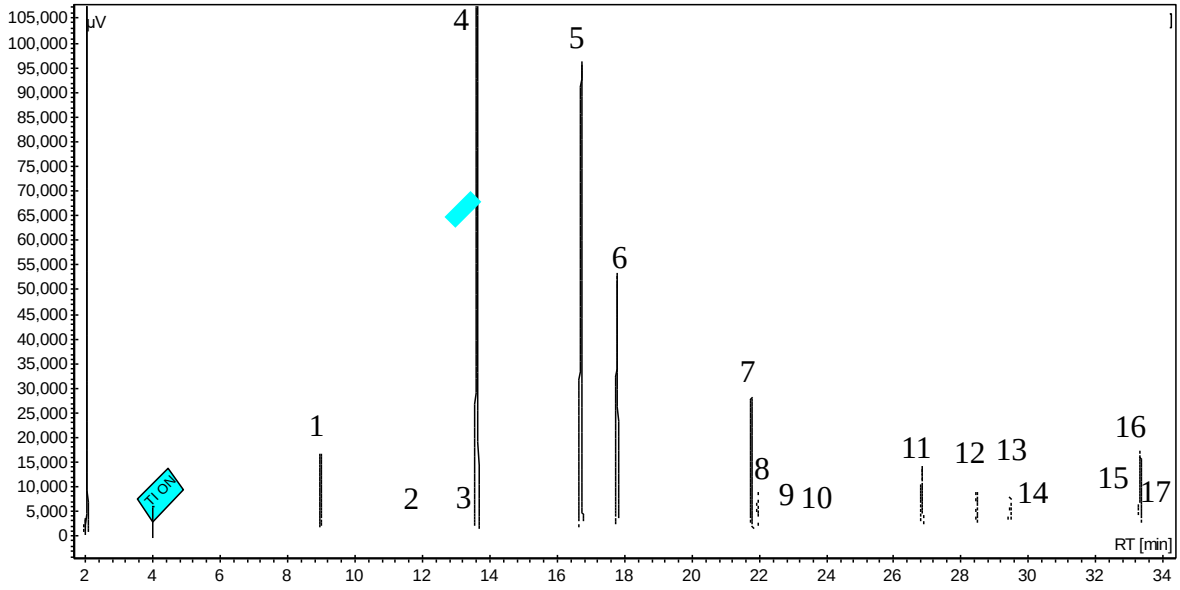

TABLE 2CHROMATOGRAPHIC PROFILE FOR NON-POLAR COLUMN

Peak No.	Component
1	Limonene
2	Linalool
3	Isopulegol
4	Citronellal
5	Citronellol
6	Geraniol
7	Citronellyl acetate
8	Eugenol
9	Geranyl Acetate
10	Beta Elemene
11	Germacrene D
12	Delta Cadinene
13	Elemol
14	Caryophyllene oxide

15	Tau-muurolol
16	Alpha cadinol
17	Bulnesol

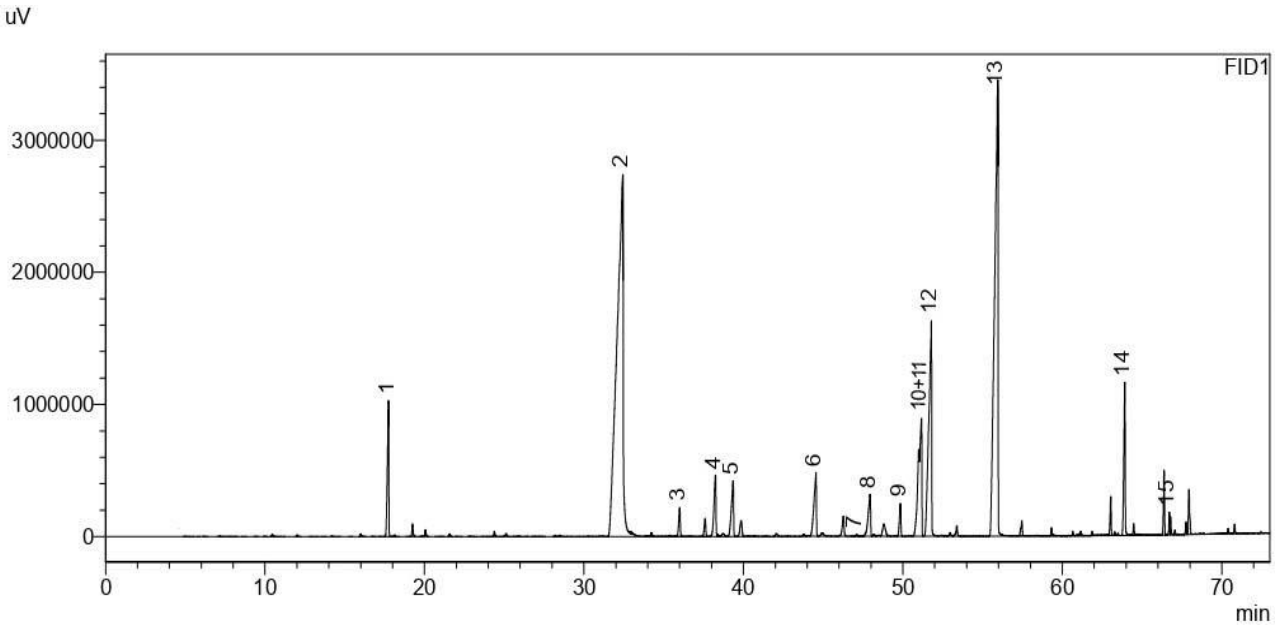

FIG. 2 – TYPICAL CHROMATOGRAM ACQUIRED ON A POLAR COLUMN

TABLE 3CHROMATOGRAPHIC PROFILE FOR POLAR COLUMN

Peak No.	**Component**
1	Limonene
2	Citronellal
3	Linalool
4	Isopulegol
5	Beta Elemene
6	Citronellyl Acetate
7	Neral
8	Germacrene D
9	Geranial
10+11	Geranyl Acetate + Delta
12	Citronellol
13	Geraniol

14	Elemol
15	Eugenol

ANNEX B

[*Clause* 4.4 *Table* 1, *Sl. No.* (ix)]

DETERMINATION OF STEAM DISTILLATION RESIDUE

B-1 REAGENTS

B-1.1 Petroleum Ether– double distilled, having boiling point range 40 to 50°C.

B-1.2 Sodium Sulphate – anhydrous *(see* IS255).

B-2 PROCEDURE

B-2.1 Place 5 g of the material, accurately weighed to the nearest mg, in a 500 ml distilling flask. Distil the material with steam under atmospheric pressure for at least 3 hours. Transfer the mixture of water and residue to a separator, rinsing the flask with two successive 20 ml portions of petroleum ether. Add the rinsing to the separator and shake vigorously for one minute. Allow to separate for 30 minutes or more, reject the aqueous layer, transfer the petroleum ether solution to a flask together with sodium sulphate.

B-2.2 Filter the petroleum ether solution through a filter paper in a previously weighed saponification flask containing some pumice stone. Rinse the flask and filter paper once more with some ether.

Gently distil off or evaporate the petroleum ether and dry the residue to constant mass within 0.01 g, at 80 ± 1°C.

B-3 CALCULATION

Steam distillation residue. Percent by mass = $\frac{M_1 \times 100}{M_2}$

where

M_1 = mass in g of the residue, and

M_2= mass in g of the sample taken for the test.