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IS 13663 (1993): Chilli Oleoresin [FAD 9: Spices and Condiments]



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भारतीय मानक

तेलीयि राल मिर्च – विशिष्टि

Indian Standard

CHILLI OLEORESIN — SPECIFICATION

UDC 664.521.1

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Spices and Condiments Sectional Committee had been approved by the Food and Agriculture Division Council.

The modern usage of spices in food industries in many overseas countries is in the form of oleoresins. Chilli oleoresin is also used for curing chewing tobacco and as counter irritant in pain balms.

Chilli oleoresin is obtained by the extraction of chillies (the fruit of red pepper, *Capsicum annum* L. or *Capsicum frutescens* L.) with approved food grade solvent and subsequent careful removal of the solvent by distillation. Besides intense pungency due to capsaicin and small quantities of allied alkaloids, the chilli oleoresin will have dark red colour due to carotenoid pigments. Colour and pungency are important from the point of view of quality of the product.

Colour is determined by instrumental analysis. For estimation of capsaicin content two methods namely TLC and Paper chromatography have been included. Also a method based on sensory evaluation is given for estimation of pungency.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the results 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 value in this standard.

Indian Standard

CHILLI OLEORESIN — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for chilli oleoresin.

2 TERMINOLOGY

For the purpose of this standard, definitions given under 2 of IS 326 : 1968 shall apply.

3 REQUIREMENTS

3.1 Description

3.1.1 The chilli oleoresin shall be obtained by solvent extraction of the dried ripe fruits of *Capsicum annuum* L or *Capsicum frutescens* L, with subsequent removal of solvent.

3.1.2 The material shall be a red viscous liquid with the characteristic odour of chilli, high pungency and deep red colour. The material shall be free from rancidity and off-flavour.

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

4 PACKING

4.1 The material shall be supplied in tightly closed glass, pure aluminium or tinfoil containers which shall be nearly full.

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

5 MARKING

5.1 The container shall be marked with the following:

- a) Name of the material;
- b) Manufacturer's name and trade mark, if any; and
- c) Net mass of the material when packed.

6 SAMPLING

6.1 Representative samples, of the material, sufficient to give a composite sample for triplicate determinations, shall be drawn from the containers selected from the lot as prescribed under 3 of IS 326 : 1968.

6.2 Tests for all the characteristics shall be conducted in the composite sample.

7 TESTS

7.1 Tests shall be conducted as prescribed in col 4 and 5 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' mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Chilli Oleoresin

(Clause 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex of this Standard	Appendix of IS 5832 : 1975
(1)	(2)	(3)	(4)	(5)
i)	Colour value	4 000-20 000	A	—
ii)	Scoville heat units, <i>Min</i>	240 000	B	—
iii)	Capsaicin content, percent by mass, <i>Min</i>	1.5	C	—
iv)	Residual solvent, mg/kg, <i>Max</i> :	—	—	C
	a) Hexane	25		
	b) Acetone/ethylene dichloride/trichloroethylene	30		
	c) Methanol/isopropanol	30		

ANNEX A

[Table 1, Item (i)]

DETERMINATION OF COLOUR VALUE

A-1 OUTLINE OF THE METHODS

To make the estimation completely objective, instrumental analysis is employed. Colour of a specified dilution is estimated at 458 nm, the absorbance reading multiplied by a dilution factor and the result expressed as Nesslerimetric colour value.

A-2 APPARATUS

A-2.1 100-ml Volumetric Flasks

A-2.2 1-ml Pipette

A-2.3 Spectrophotometer or a colorimeter with adjustable wave length

A-3 REAGENTS

A-3.1 Acetone

A-4 PROCEDURE

A-4.1 Weigh 1.0 g of oleoresin into a 100-ml volumetric flask, and make up the volume with acetone. Pipette 1.0 ml of this solution into a second 100-ml volumetric flask and make up the volume with acetone.

A-4.2 Using a tungsten lamp source and acetone as the blank, take the absorbance of the 0.01 percent solution of oleoresin at 458 nm. Multiply this reading by 61000 to obtain the colour value.

ANNEX B

[Table 1, Item (ii)]

DETERMINATION OF SCOVILLE HEAT UNIT

B-1 OUTLINE OF THE METHOD

This is a method based on sensory evaluation. Oleoresin solutions variably diluted with sugar solution are tasted in increasing concentration. The highest dilution when a pungency is just detected is taken as a measure of the heat value.

B-2 REAGENTS

B-2.1 5 Percent Sugar Solution

B-2.2 95 Percent Alcohol

B-3 PROCEDURE

B-3.1 Weigh accurately 0.2 g of the oleoresin into a 50-ml volumetric flask. Add alcohol to the volume mark and shake the contents and allow to settle.

B-3.2 Add 0.15 ml of alcoholic solution to 140 ml sugar solution. Swallow after thorough

shaking, 5 ml of this solution. A bite or stinging sensation in the throat which is just perceptible at this dilution is equal to 240 000 scoville units. If bite is very strong, a further dilution of this solution as given below is necessary for a perceptible pungent sensation:

- a) 15 ml of sugar solution
30 ml solution (B-3.2) = 360 000
Scoville units
- b) 20 ml of sugar solution
20 ml solution (B-3.2) = 480 000
Scoville units
- c) 30 ml sugar solution
15 ml solution (B-3.2) = 720 000
Scoville units

The panel for evaluation shall consist of 5 members, and 3 members out of 5 shall find the solution to be pungent.

ANNEX C

[Table 1, Item (iii)]

DETERMINATION OF CAPSAICIN CONTENT

C-1 GENERAL

The pungency of chilli is due to capsaicin and traces of allied chemicals. Pungency can be objectively evaluated by estimating the capsaicin content. For this purpose, two methods, namely, TLC and paper chromatography are equally suitable.

C-2 TLC METHOD**C-2.1 Outline of the Method**

The capsaicin and allied constituents may be separated from other constituents by thin layer chromatography (TLC) on silica gel. The pungent principle can react with Folin-Dennis reagent to give a blue complex which may be estimated colorimetrically.

C-2.2 Apparatus**C-2.2.1 Complete TLC Set****C-2.2.2 Spectrophotometer or Colorimeter**

Capable of measuring in the region of 660 to 725 nm.

C-2.2.3 Measuring Jar with Stopper — 10 ml.**C-2.2.4 Test-Tubes****C-2.2.5 Graduated 10-ml Pipettes — with 0.1 ml graduation.****C-2.3 Reagents****C-2.3.1 Silica Gel (TLC Grade)****C-2.3.2 Developing Solvent**

Mixture of 80 ml of benzene and 5 ml of methanol.

C-2.3.3 Phosphomolybdic-Phosphotungstic Acid (Folin-Dennis Reagent)

Reflux for 2 hours, 750 ml water, 100 g sodium tungstate, 20 g phosphomolybdic acid and 50 ml phosphoric acid. Cool the mixture and dilute to 1 000 ml with water.

C-2.3.4 Vanillin — analytical grade.**C-2.3.5 Benzene — TLC grade****C-2.3.6 Methanol — TLC grade****C-2.4 Procedure****C-2.4.1 Preparation of TLC Plates**

Mix 30 g silica gel containing calcium sulphate as binder. Mix with 60 ml distilled water. Pour the slurry into a TLC spreader adjusted to a thickness of 150 μ m and spread over 5 to 6 glass plates of 20 \times 20 cm. Air dry the plates for 4 to 5 hours, later activate by drying in an oven at 100-105°C for 30 minutes and store in a desiccator.

C-2.4.2 Estimation

C-2.4.2.1 Weigh 5 g oleoresin in a 10-ml stoppered measuring jar and make up the volume with acetone. Spot 10 μ l (5 to 20 μ l depending upon the capsaicin content) of this solution on TLC plate and develop with developing solvent in an all glass chamber which has been thoroughly saturated with the vapour of the solvent. Expose the plate for about 30 minutes to free it of the solvent and lightly spray with phosphomolybdic-phosphotungstic reagent. Mark the clear blue capsaicin spot (R_f 0.16) with a small stainless steel scoop or spatula in the form of a circle enclosing the spot as well as an area 0.25 cm beyond the spot. Scoop out the silica gel containing capsaicin in the marked area into a clean butter paper and transfer into a test tube. Add 3.5 ml distilled water and shake well. Pipette out 0.5 ml phosphomolybdic-phosphotungstic reagent into the tube and mix well. After 3 minutes add 1 ml saturated aqueous sodium carbonate solution. Mix thoroughly for 5 minutes and set aside for 1 hour.

C-2.4.2.2 Prepare a reagent blank, using silica gel layers from a blank area in the plate. Centrifuge the tubes (or filter) at the end of one hour to separate solids. Read optical density in spectrophotometer at 725 nm or in a colorimeter using a glass filter in 660 to 690 nm region.

C-2.4.3 Determine the amount of X capsaicin in the spot from a standard curve of concentration of pure vanillin versus optical density. Multiply by 2 (a factor) to correct for the difference in the molecular mass of capsaicin and vanillin.

C-2.4.4 Preparation of Standard Curve

Different volumes of a standard aqueous solution of vanillin are reached with Folin-

Dennis reagent and the optical density is determined.

NOTE — The standard aqueous solution of vanillin should contain 0.01 mg/ml. 1 to 3.5 ml of this solution react with 0.5 ml Folin-Dennis reagent and 1 ml saturated aqueous sodium carbonate solution, keeping the total volume to 5 ml by addition of distilled water.

C-2.4.5 Draw a standard curve plotting mass of vanillin against optical density.

C-2.5 Calculation

$$\text{Capsaicin content, percent by mass} = \frac{X \times 1\,000 \times 200}{r}$$

where

X = the number of g capsaicin (vanillin $\times 2$) in the spot, and

r = the number of μl of 50 percent acetone solution of oleoresin.

C-3 PAPER CHROMATOGRAPHY METHOD

C-3.1 Outline of the Method

The pungent principles may be separated from other constituents by paper chromatography and they can react with Gibbs reagent to give a blue complex which may be estimated colorimetrically.

C-3.2 Apparatus

C-3.2.1 Chamber — for ascending paper strip chromatography.

C-3.2.2 Spectrophotometer or Colorimeter — with narrow band filter.

C-3.3 Reagents

C-3.3.1 Ethyl Acetate — Solvent of capsaicin or dilution of total extracts.

C-3.3.2 Methanol — Analytical reagent grade

C-3.3.3 Boric Acid — Analytical reagent grade

C-3.3.4 Potassium Chloride

C-3.3.5 Buffer Solution

A solution containing 3.1 g boric acid and 3.7 g potassium chloride in 100 ml distilled water adjusted to pH 9.6 with 1 N sodium hydroxide.

C-3.3.6 Methanol-Buffer Solution

pH 9.6 (60+40 v/v) as developing solvent.

C-3.3.7 Gibbs Reagent

0.1 percent solution of 2, 6-dichloro-p-benzoquinon-4-chlorimine in acetone as the chromogenic reagent.

C-3.4 Procedure

C-3.4.1 Dissolve the oleoresin or extractives in ethyl acetate to give a solution containing about 2.5 g/l of capsaicin. Apply an amount of the solution containing 10-50 μg capsaicin (that is 4 to 20 μl) as a thin streak covering the entire width of a 2 \times 20 cm strip of Whatman No. 3 filter paper. Develop by ascending chromatography in methanol-buffer solvent until the solvent front has moved up to about 15 cm, usually in about 1 hour. Dry the strip in air.

C-3.4.2 Pass the dried paper strip uniformly through Gibbs reagent and dry in air. Capsaicin becomes faintly visible as a blue spot near the solvent front. Develop the colour further by spraying with buffer solution, lightly on both sides of the paper. Transfer the strip to a dark cupboard for 30 minutes for full development of colour and drying.

C-3.4.3 Excise the blue spot and elute with methanol in the dark. Make up the eluted colour to a convenient volume (5-10 ml) and measure at 615 nm in a spectrophotometer against a reagent blank eluted from the corresponding area of a strip run simultaneously without the sample. Calculate the percentage of capsaicin from a standard curve constructed as given in **C-3.4.5** or by using the formula:

$$E = \frac{\text{1 percent}}{\text{1 cm}} = 640 \text{ at } 615 \text{ nm.}$$

C-3.4.4 Prepare the oleoresin or total extract of the sample by Soxhlet extraction of powdered capsicum with a suitable solvent (ethyl acetate) for 2.5-3 hours. After removal of solvent, weight the residue and proceed as in **C-3.4**.

C-3.4.5 Prepare a solution of pure capsaicin in ethyl acetate (2 g/l), spot 10, 20, 30, 40 and 50 μg of capsaicin (5-25 μl) on strips of Whatman No. 3 filter paper, develop, visualize the capsaicin spot, and elute. Construct the standard curve and

$$E = \frac{\text{1 percent}}{\text{1 cm}} \text{ for pure capsaicin.}$$

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AMENDMENT NO. 1 NOVEMBER 1994
TO
IS 13663 : 1993 CHILLI OLEORESIN —
SPECIFICATION

(*Page 1, clause 2*) — Substitute the following for the existing matter:

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
326 (Part 1) : 1984	Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
5832 : 1984	Oleoresin black pepper (<i>second revision</i>)

(*Page 1, clause 6.1, last line*) — Substitute 'in IS 326 (Part 1) : 1984' for 'under 3 of IS 326 : 1968'.

(*Page 1, Table 1, col 5, heading*) — Substitute 'IS 5832 : 1984' for 'IS 5832 : 1975'.

AMENDMENT NO. 2 DECEMBER 1998
TO
IS 13663 : 1993 CHILLI OLEORESIN —
SPECIFICATION

(Page 1, clause 3.1.2) — Insert a new clause after 3.1.2 as under:

'3.1.3 It shall be free from added colouring matter.'

(FAD 9)

Reprography Unit, BIS, New Delhi, India