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IS 11909 (1986): Sweet Orange Oil, Cold Pressed, Food Grade
[FAD 8: Food Additives]

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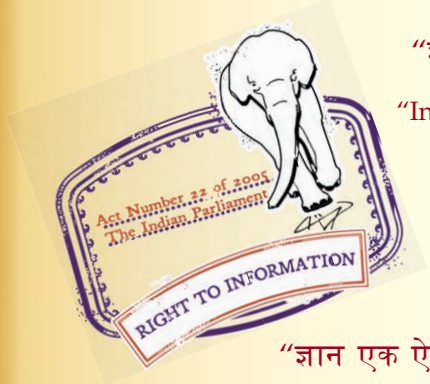

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"Knowledge is such a treasure which cannot be stolen"

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IS : 11909 - 1986
(Reaffirmed 2003)

Indian Standard

SPECIFICATION FOR SWEET ORANGE OIL, COLD PRESSED, FOOD GRADE

UDC 664·099 : 665·526·444

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

Gr 3

May 1987

Indian Standard

SPECIFICATION FOR SWEET ORANGE OIL, COLD PRESSED, FOOD GRADE

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*Dr T. S. Santhanakrishnan acted as chairman at the meeting in which this document was finalized.

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Indian Standard

SPECIFICATION FOR SWEET ORANGE OIL, COLD PRESSED, FOOD GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 November 1986, after the draft finalized by the Food Additives Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As certain impurities in these substances could be harmful, it is necessary to have a strict quality control of these food additives. A series of standards is, therefore, being prepared to cover purity and identification of these substances. These standards would help in checking purity which requires to be checked at the stage of manufacture for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

0.3 Sweet orange oil, cold pressed, is the volatile oil obtained by expression from the fresh peel of the ripe fruit of *Citrus Sinensis* L. Osbeck (Fam Rutaceae). It is an intensely yellow, orange or deep orange liquid, having the characteristic odour and taste of the outer part of fresh, sweet orange peel.

0.3.1 Sweet orange oil, cold pressed, is widely used as a flavouring agent in foods.

0.4 In the preparation of this standard considerable assistance has been derived from Food Chemical Codex, Pub. National Academy of Sciences and National Research Council, Washington DC, USA.

0.5 A separate Indian Standard for oil of Mandarin Orange (*Citrus reticulata*), cold pressed (IS : 6617-1972) used in soap, confectionery, perfumery and beverage industry has already been published.

0.6 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*. 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

1.1 This standard prescribes the requirements and the methods of sampling and test for sweet orange oil, cold pressed, food grade.

2. REQUIREMENTS

2.1 Description — Sweet orange oil, cold pressed is an intensely yellow, orange or deep orange liquid, having the characteristic odour and taste of the outer part of fresh, sweet orange peel.

2.2 Solubility — Sweet orange oil, cold pressed shall be miscible with anhydrous alcohol and with carbon disulphide. It shall be soluble in glacial acetic acid.

2.3 Angular Rotation — The angular rotation at 25°C in neat oil in a 100-mm tube shall be between +94° and +99°.

2.4 Refractive Index — The refractive index at 20°C shall be between 1·472 and 1·474.

2.5 Specific Gravity — The specific gravity at 25°/25°C shall be between 0·842 and 0·846.

2.6 The material shall also conform to the requirements given in Table 1.

3. PACKING, STORAGE AND MARKING

3.1 Packing — The material shall be filled in food lacquered M.S. drums or other suitable containers with as little air space as possible. The containers shall be such as to preclude air contamination of the containers with metal or other impurities.

3.2 Storage — The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR SWEET ORANGE OIL, COLD PRESSED, FOOD GRADE

(*Clause* 2.6)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of This Standard	Clause of IS : 1699-1974*
(1)	(2)	(3)	(4)	(5)
i)	Aldehydes (as $C_{10}H_{20}O$) percent by mass	1·2 to 2·5	A	—
ii)	Foreign oils	To pass test	B	—
iii)	Washed citrus oils	To pass test	C	—
iv)	Lead (as Pb), mg/kg, *Max*	10	—	9
v)	Arsenic (as As), mg/kg, *Max*	3	—	10
vi)	Heavy metals (as Pb), mg/kg, *Max*	40	D	—

*Methods of sampling and test for food colours (*first revision*).

3.3 Marking — Each container shall be marked legibly to give the following information:

a) Name of the material including the words, 'food grade';

b) Name and address of the manufacturer;

c) Minimum net mass of contents;

d) Batch or code number; and

e) Date of manufacture.

3.3.1 The container 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 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in **3** of IS : 1699-1974*.

*Methods of sampling and test for food colours (*first revision*).

5. TESTS

5.1 Tests shall be carried out by the methods specified in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

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

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ALDEHYDES

A-1. REAGENTS

A-1.1 Hydroxylamine Hydrochloride Solution — Dissolve 50 grams of hydroxylamine hydrochloride (preferably recrystallized before using) in 90 ml of water and dilute to 1 000 ml with aldehyde-free alcohol. Adjust the solution to a *p*H of about 3·4 with 0·5 N alcoholic potassium hydroxide.

A-1.2 Bromophenol Blue Test Solution — Dissolve 100 mg of bromophenol blue in 100 ml of dilute alcohol (1 in 2) and filter if necessary. For *p*H determinations, dissolve 100 mg in 3·2 ml of 0·05 N sodium hydroxide, and dilute with carbon dioxide-free water to 200 ml.

A-2. PROCEDURE

A-2.1 Weigh accurately 10 ml of sample and transfer into a 125-ml Erlenmeyer flask. Add 30·0 ml of the hydroxylamine hydrochloride solution and mix thoroughly. Allow the mixture to stand at room temperature for 10 minutes with occasional shaking. Titrate the liberated hydrochloride acid with 0·5 N alcoholic potassium hydroxide to a greenish-yellow endpoint using bromophenol blue test solution as indicator or to a *p*H of 3·4 using a suitable *p*H meter. Perform a blank determination simultaneously with the sample determination.

*Specification for water for general laboratory use (*second revision*).

A-3. CALCULATIONS

$$\text{Aldehydes, percent by mass} = \frac{V - V_1 (78{\cdot}14 \times 100)}{M}$$

where

V = ml of 0·5 N alcoholic potassium hydroxide consumed in titration of the sample;

V_1 = ml of 0·5 N alcoholic potassium hydroxide consumed in titration of the blank; and

M = mass of the sample in mg.

APPENDIX B

[*Table* 1, *Item* (ii)]

TEST FOR FOREIGN OILS

B-1. PROCEDURE

B-1.1 Transfer 50·0 ml of the sweet orange oil to a Ladenburg flask having 4 bulbs of approximate diameters 6 cm, 3·5 cm, 3·0 cm and 2·5 cm respectively in ascending order. The distance from the bottom of the flask to the side arm is 20 cm. Distil the oil at the rate of 1 drop per second until the distillate measures 5 ml. The angular rotation of the distillate shall not differ from that of the original oil by more than 2 degrees and the refractive index of the distillate shall not be less than 0·000 5 and not more than 0·001 8 lower than that of the original oil at 20°C.

APPENDIX C

[*Table* 1, *Item* (iii)]

TEST FOR WASHED CITRUS OILS

C-1. PROCEDURE

C-1.1 Evaporate 3 ml of the sweet orange oil in a tared glass crystallizing dish on a steam bath for 5 hours. Continue heating at 105°C for 2 hours. Cool in a desiccator and weigh. Not less than 43 mg of residue remains.

C-1.2 A 1-ml sample does not form a clear solution with 2 ml of 89·0 percent alcohol.

APPENDIX D

[*Table* 1, *Item* (vi)]

DETERMINATION OF HEAVY METALS

D-1. REAGENTS

D-1.1 Ammonia Solution — Dilute 400 ml of ammonium hydroxide (28 percent) to 1 000 ml with water.

D-1.2 Hydrochloric Acid — 10 percent.

D-1.3 Lead Nitrate Stock Solution — Dissolve 159·8 mg of lead nitrate in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1 000 ml and mix. Prepare and store the solution in lead free glass containers.

D-1.4 Standard Lead Solution — Dilute 10 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of 10 μg of lead ion (Pb). Prepare the solution on the day of use.

D-1.5 Nitric Acid — 10 percent (*v/v*).

D-1.6 Sulphuric Acid — 94·5 to 95·5 percent (*v/v*).

D-1.7 Acetic Acid — 6 percent (*m/v*).

D-1.8 Hydrogen Sulphide — A saturated solution of hydrogen sulphide made by passing H_2S in cold water.

D-2. PROCEDURE

D-2.1 Solution A — Take 2 ml of the standard lead solution in a 50-ml Nessler tube and add 23 ml of water. Adjust the *p*H to between 3·0 and 4·0 by addition of acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

D-2.2 Solution B — Place 500 mg of the sample, accurately weighed in a suitable crucible. Add sufficient nitric acid to wet the sample, and carefully ionite at a low temperature until thoroughly charred, covering the crucible loosely with a suitable lid during ignition. After the substance is thoroughly carbonized, add 2 ml of nitric acid and .5 drops of sulphuric acid and cautiously heat until white fumes are evolved. Then ignite, preferably in a muffle furnace, at 500 to 600°C until the carbon is all burnt off. Cool, add 4 ml of dilute hydrochloric acid, cover and digest on a steam bath for 10 to 15 minutes. Uncover and slowly evaporate on a steam-bath to dryness. Moisten the residue with one drop of hydrochloric acid, add 10 ml of hot water and digest for 2 minutes. Add, dropwise, ammonia solution until the solution is just

alkaline to litmus paper. Dilute with water to 25 ml and adjust the *p*H to between 3·0 and 4·0 (*p*H indicator paper) by the addition of dilute acetic acid. Filter if necessary. Wash the crucible and the filter with 10 ml of water. Transfer to a 50-ml Nessler tube. Dilute the combined filtrate and washing with water to 40 ml and mix.

D-2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide. Mix and allow to stand for 45 minutes and view down over a white surface. The colour of solution *B* shall not be darker than that of solution *A*.

(*Continued from page* 2)

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