SRI LANKA STANDARD 435 : 1978
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SPECIFICATION FOR
OIL OF CARDAMOM

BUREAU OF CEYLON STANDARDS

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SLS 435 : 1978

Gr. 4

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Sri Lanka.

SRI LANKA STANDARD

SPECIFICATION FOR OIL OF CARDAMOM

FOREWORD

This Sri Lanka Standard Specification has been prepared by the Drafting Committee of the Bureau on Essential Oils. It was approved by the Agricultural and Chemicals Divisional Committee of the Bureau of Ceylon Standards and was authorised for adoption and publication by the Council of the Bureau on 1978-03-14.

Oil of cardamoms is used in the food and perfumery industries.

The standard values are given in SI units.

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 the test, shall be rounded off in accordance with CS 102. The number of figures to be retained in the rounded off value, shall be the same as that of the specified value in this standard.

This standard makes reference to the following Standards:

CS 102 Presentation of numerical values

SLS 210 Method for the preparation of test samples of essential oils

SLS 211 Methods for labelling and marking of containers for essential oils

SLS 212 Methods for packing of essential oils

SLS 213 Methods for sampling of essential oils

In the preparation of the standard, the assistance derived from the publications of the International Organization for Standardization (ISO) and the Essential Oil Association of U.S.A. is acknowledged.

1 SCOPE

This standard prescribes requirements and methods of test for oil of cardamom.

2 DEFINITION

The oil shall be the product obtained by steam distillation of the fruits of *Elettaria cardamomum* (L) Maten. var, miniscula Burkhill* [*Elettaria repens*' (Sonner) Baill**]

3 REQUIREMENTS

3.1 Description

The oil shall be colourless to pale yellow in colour. It shall have the characteristic aromatic penetrating odour of cardamoms and a strongly aromatic spicy taste.

3.2 Relative density at 30 ^{o}C/30 ^{o}C

The relative density of the oil at 30 ^{o}C when determined by the method described in Appendix A shall not be less than 0.912 and not more than 0.936.

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*E. Guenther The Essential Oils, Volume 5, p.96, Van Nostrand, New York.

**B.A.Abeywickrema, Ceylon J. Sci (Biol.Sci)2, 145 (1959).

3.3 Optical rotation

The optical rotation of the oil at 30 °C when determined by the method described in Appendix B shall be within the range + 22° to + 41°.

3.4 Refractive index

The refractive index of the oil at 30 °C when determined by the method described in Appendix C shall be not less than 1.4620 and not more than 1.4680.

3.5 Solubility in ethanol

One volume of oil shall be soluble in 2 to 5 volumes of 70 per cent (v/v) ethanol at 30 °C to give a clear solution when tested by the method described in Appendix D.

3.6 Ester value

The ester value of the oil when determined by the method described in Appendix E shall be not less than 90 and not greater than 150.

4 PACKAGING AND MARKING

These shall be shipped preferably in glass containers or in any other suitable containers as agreed to between the buyer and the seller. SLS 212 and SLS 211 are broadly applicable to essential oils.

5 SAMPLING AND SIZE OF SAMPLE

A representative sample or samples as required, each measuring not less than 50 ml shall be taken preferably from original containers or from the bulk, for the purpose of examination. The samples shall be placed in clean, dry, air-tight, non-absorbent containers on which the sample has no action. The containers shall be of such size that they are nearly filled by the sample.

Each container so filled shall be marked with full details and date of sampling and shall be protected from light. **SLS** 213 and **SLS** 210 are broadly applicable to all essential oils.

6 STORAGE

The oil shall be stored in air-tight full containers away from light.

APPENDIX A

DETERMINATION OF RELATIVE DENSITY

A.1 GENERAL

The relative density of the material shall be expressed as the ratio of the density of oil at 30 °C to that of distilled water at the same temperature.

A.1.1 Even though the specified temperature for the relative density is 30 °C, the determination may be conducted within 30 ± 3 °C. Usually the relative density decreases with increase in temperature, and so the value for 30 °C is calculated as given below:

a) If the temperature of testing is higher than 30 °C to the value found, add for each degree celsius of difference between the two temperatures, the correction factor 0.000 64.

b) If the temperature of testing is lower than 30 °C from the value found, subtract for each degree celsius of the difference between the two temperatures, the correction factor 0.000 64.

ERRATUM

SLS 435:1978 SPECIFICATION FOR OIL OF CARDAMOM

Page 7 - Substitute the Clause Number **A.2** to the existing Clause Number **A.1.2.**

A.1.2 Relative density may be determined with a specific gravity bottle or a pyknometer and in case of dispute, the determination shall be made at 30 °C.

A.3 PROCEDURE

A.3.1 With pyknometer or specific gravity bottle

Clean the pyknometer or specific gravity bottle of at least 5-ml capacity, using a saturated solution of chromium trioxide in concentrated sulphuric acid, rinse thoroughly with distilled water, dry and allow to stand for at least three hours and weigh. Fill the pyknometer or specific gravity bottle with recently boiled distilled water which has been cooled to a temperature three degrees lower than the test temperature. Keep the pyknometer or specific gravity bottle with contents in a water bath at the test temperature for 30 minutes. Adjust the level of the water up to the mark, removing any excess with clean filter paper or cloth, and put the ground glass cap in place. Remove the pyknometer or specific gravity bottle from the water bath, dry carefully with a clean cloth, permit it to stand for 30 minutes and weigh accurately. Empty the pyknometer or specific gravity bottle, rinse several times with ethanol and finally with ether. Remove the ether vapours with the aid of an air blast and permit the pyknometer or specific gravity bottle to dry thoroughly. Fill the clean, dried pyknometer or specific gravity bottle with the cardamom oil previously cooled to a temperature 3 degrees lower than the test temperature. Following the same procedure as above, place the pyknometer or specific gravity bottle in a water bath and permit it to warm slowly to the test temperature. As before adjust the material to the proper level, put the cap in place and wipe the specific gravity bottle or pyknometer dry. Accurately weigh after 30 minutes.

A.3.2 The ratio of the mass of the oil to the mass of the water contained in the pyknometer or specific gravity

bottle gives the relative density (S) of the oil at the test temperature ($t\ ^{o}C$)

$$S_{t\ ^{o}C/t\ ^{o}C} = \frac{m_1}{m_o}$$

where

m_o = mass of water

m_1 = mass of oil.

A.4 REPORT

The temperature at which the test was carried out and the correction factor, if used, shall be stated in the report.

APPENDIX B

DETERMINATION OF OPTICAL ROTATION

B.1 PRINCIPLE

The optical rotation of the material is the angle in degrees through which the plane of polarization is turned when plane-polarized sodium light is passed through a layer of oil, 100 mm in thickness.

B.2 APPARATUS

B.2.1 *Polarimeter,* of suitable type with a precision of $\pm\ 0.03^{o}$. It should give a reading of 0^{o} and also 180^{o} with distilled water when properly adjusted.

B.2.2 *Light source :* Any apparatus giving monochromatic light from sodium vapour lamp.

B.2.3 *Polarimeter tubes,* 100 ± 0.05 mm.

B.3 PROCEDURE

B.3.1 Calibration of the polarimeter

Check the apparatus by finding out the optical rotation of a solution of sucrose containing 26 grams of sucrose in 100 ml of solution at a temperature of 30 °C and the optical rotation should be + 25.9°.

B.3.2 Switch the light source on, and wait until full luminosity is obtained. Fill the polarimeter tubes with the material at 30 ± 1 °C and ensure the absence of air bubbles.Place the tube in the polarimeter and read the dextrorotatory (+) or laevorotatory (-) optical rotation of the material on the scale of the instrument. Conduct the determination preferably in a dark room. Record the results as the average of at least three readings which should agree within 0.08° and rounded to first decimal place.

B.3.3 If the material is found to be excessively coloured, shake a portion of the sample of the material with powdered tartaric acid for about five minutes and filter. This treatment improves the colour of the material.

APPENDIX C

DETERMINATION OF REFRACTIVE INDEX

C.1 PRINCIPLE

The refractive index of the material is the ratio of the sine of the angle of incidence to the sine of the angle of refraction when a ray of light of wavelength

589.3 nm (the mean of the D lines of sodium) passes from air into the material.

The notation is refractive index n_D^t, t being the temperature, (^oC) at which the determination is made.

C.2 PROCEDURE

Determine the refractive index in a standard instrument* employing the principle of the critical angle, using diffused daylight or any convenient artificial light as illuminant. Maintain the prisms at the specified temperature and allow the material to stay on the prism surface for a couple of minutes to attain the required temperature. Take a second reading after the lapse of a few minutes.

C.2.1 A temperature of 30 ^oC is recommended. Carry out the determination at or as near as possible to the temperature specified.

C.2.2 Since moisture in the air may condense on the cooled prisms, great care shall be exercised when determining refractive indices during hot, humid weather. Occasionally, the instrument should be checked by means of the quartz plate that accompanies it, using monobromonaphthalene, or if such a plate is not available, by means of distilled water at 30 ^oC. The refractive index of distilled water at 30 ^oC is 1.332 00.

C.2.3 If, for any reason, the refractive index cannot be determined at the specified temperature, apply the correction factor of 0.000 38 per degree celsius. If the refractive index is determined at a temperature above the specified temperature, add the appropriate correction; if determined below the specified temperature, subtract the appropriate correction.

**A suitable type of instrument for this purpose is the Abbe refractometer.*

C.2.4 Report

Report the refractive index at 30 °C as a number correct to four decimal places.

APPENDIX D

DETERMINATION OF SOLUBILITY IN ETHANOL

D.1 SOLVENT

Reagent quality ethanol of 70 per cent v/v at 30 °C shall be used. Prepare by weighing ethanol (95 per cent by volume) and distilled water in the proportion of 676:324 and mix them thoroughly. The strength of the ethanol at this temperature shall be checked.

D.2 APPARATUS

A 10-ml glass stoppered cylinder, graduated at 0.1-ml intervals.

D.3 PROCEDURE

Introduce exactly 1 ml of the oil into the cylinder and add, slowly and in small proportions, the ethanol, shaking the contents of the cylinder thoroughly after each addition. When a clear solution is first obtained record the number of volumes of ethanol added. Continue the addition of ethanol until the 6-ml mark on the cylinder has been reached.

D.4 REPORT

Report the number of volumes of 70 per cent (v/v) ethanol at 30 °C required to obtain a clear solution when it is mixed with 1 ml of oil.

APPENDIX E

DETERMINATION OF ESTER VALUE

E.1 DEFINITION

The ester value of an essential oil is the mass of potassium hydroxide, in milligrams, required to neutralize the acids liberated by the hydrolysis of the esters present in 1 gram of the oil.

E.2 REAGENTS

The reagents used shall be of a recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used throughout.

E.2.1 *Ethanol,* 95 per cent (v/v).

E.2.2 *Potassium hydroxide,* approximately 0.5 N ethanolic solution. Prepare by dissolving 8.5 g of potassium hydroxide in 250 ml of the ethanol. Allow to stand and decant or filter the clear liquid.

E.2.3 *Potassium hydroxide,* 0.1 N ethanolic solution.

E.2.4 *Hydrochloric acid,* 0.5 N solution.

E.2.5 *Phenolphthalein indicator,* 0.2 per cent solution in ethanol, 60 per cent (v/v).

E.3 APPARATUS

E.3.1 *Flask,* 250-ml capacity, made of chemically resistant glass and with a neck terminating in a ground socket.

E.3.2 *Reflux condenser,* having a ground cone for attachment to the flask.

E.4 PROCEDURE

Weigh into a saponification flask, to an accuracy of 1 mg, 4.5 g to 5.0 g of the oil. Add 5 ml of the ethanol and neutralize with the 0.1 N ethanolic potassium hydroxide solution, using the phenolphthalein indicator. Add to the neutralized solution 25.0 ml of the 0.5 N ethanolic potassium hydroxide solution and boil the mixture under a reflux condenser for 1 hour. Cool, add 20 ml of water and immediately titrate the excess of alkali with the 0.5 N hydrochloric acid, using an additional 0.5 ml of the phenolphthalein indicator. Make a blank determination, following the same procedure but omitting the oil, and using 5 ml of water.

Ignore any reappearance of the pink colour on standing.

E.5 CALCULATION OF ESTER VALUE

$$\text{Ester value } E = \frac{28.5 \times (V - V_1)}{m}$$

where

V = volume in millilitres, of 0.5 N hydrochloric acid required for the blank,

V_1 = volume, in millilitres, of 0.5 N hydrochloric acid required to neutralize the excess of alkali used for the hydrolysis, and

m = mass, in grams, of oil taken.

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