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SPECIFICATION FOR OIL OF PEPPER

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BUREAU OF CEYLON STANDARD

SPECIFICATION FOR OIL OF PEPPER

S.L.S. 387 : 1976

Gr. 4

BUREAU OF CEYLON STANDARDS
53, DHARMAPALA MAWATHA
COLOMBO-3.

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This Standard does not purport to include all the necessary provisions of a contract.

BUREAU OF CEYLON STANDARDS
53, DHARMAPALA MAWATHA
COLOMBO-3.

Telephone: 26055
26054
26051

Telegrams: "PRAMIKA"

SRI LANKA STANDARD SPECIFICATION FOR OIL OF PEPPER

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 7th July, 1976.

Oil of pepper is used in the food, pharmaceutical and perfumery industries.

The standard values are given in SI units. For the purpose of deciding whether a particular requirement of this standard complies with the final value observed or calculated, expressing the result of the test, shall be rounded off in accordance with C.S. 102:1971.* 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 requires reference to the following:—

CS 210 : 1973 — Method for the preparation of test samples of essential oils.

CS 211 : 1973 — Methods for labelling and marking of containers for essential oils.

CS 212 : 1973 — Method for packing of essential oils.

CS 213 : 1973 — Methods for the sampling of essential oils.

In the preparation of the standard, the assistance derived from the publications of the British Standard Institution is acknowledged.

1. SCOPE

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

* C S 102 : 1971—Presentation of Numerical Values.

2. DEFINITION

The oil shall be the product obtained by steam distillation of the dried fully mature fruits of *Piper nigrum* L.

3. REQUIREMENTS

3.1 **Description**—The oil shall be almost colourless to very pale bluish green in colour. It shall have the characteristic odour of pepper and shall have the mild flavour, lacking the pungency of the spice.

3.2 **Relative density at 32°C/30°C**—The relative density of the oil at 30°C when determined by the method described in Appendix A shall not be less than 0.868 and not more than 0.907.

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 −15° to + 4°.

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 1.480 and not more than 1.492.

3.5 **Solubility in Ethanol**—1 volume of oil shall be soluble in 3 volumes of 95 % (v/v) ethanol at 30°C 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 not be greater than 11.

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. CS 212: 1973* and CS 211 : 1973** 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

* C S 212 : 1973—Method for packing of essential oils.

** C S 211 : 1973—Methods for labelling and marking of containers for essential oils.

clean, dry, airtight, 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. CS 213: 1973* and CS 210 : 1973** are broadly applicable to all esesntial oils.

6. STORAGE

The oil shall be stored in airtight 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 a specified temperature to that of distilled water at the same temperature.

A-1.1 The buyer and the seller may, by mutual agreement, fix any convenient temperature for the determination of relative density. A temperature of 30°C is recommended unless otherwise agreed to. Usually the relative density decreases with increase in temperature, and so the value for the specified temperature is calculated as given below:

(*a*) If the temperature of testing is higher than the specified temperature to the value found, add for each degree Celsius of the difference between the two temperatures the correction factor 0.000 64.

(*b*) If the temperature of testing is lower than the specified temperature, from the value found subtract for each degree Celsius of the difference between the two temperatures, the correction factor 0.000 64.

(*c*) The specified correction factor holds good within ± 3 deg. of the specified temperature.

* C S 213 : 1973—Methods for the sampling of essential oils.
** C S 210 : 1973—Methods for the preparation of test-sample of essential oils.

A-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 the specified temperature.

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; dry and allow it to stand for at least 3 hours. Empty the pyknometer or specific gravity bottle and rinse thoroughly with distilled water. Fill the pyknometer or specific gravity bottle with recently boiled distilled water which has been cooled to a temperature 3 deg, lower than the test temperature. Keep the pyknometer or specific gravity bottle with contents in a water-bath at the test temperature or 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. Weigh accurately after standing for 30 minutes. Fill the clean, dried pyknometer or specific gravity bottle with the pepper oil previously cooled to a temperature 3 deg. 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 of the oil at the test temperature.

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 GENERAL

For the purpose of this determination, the optical rotation of the material is taken as 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°. It should give a reading of 0° and also 180° 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 $\pm$ 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 grammes 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 $\pm$ 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 5 minutes and filter. This treatment improves the colour of the material.

APPENDIX C

DETERMINATION OF REFRACTIVE INDEX

C-1 GENERAL

For the purpose of this determination, the refractive index of the material is taken as 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 mm (the mean of the D lines of sodium) passes from air into the material.

The notation is refractive index n_D^t, being the temperature, (°C) 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°C 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 monobromnaphthalene, or if such a plate is not available, by means of distilled water at 30°C. The refractive index of distilled water at 30°C is 1.332 00.

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

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.

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 95 per cent v/v at 30°C shall be used. 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 4 ml mark on the cylinder has been reached.

D-4 REPORT

Report the 1 volume sample as being soluble in 3 volumes of 95% (v/v) ethanol at 30°C if the mixture obtained by procedure D-3 is clear.

APPENDIX E

DETERMINATION OF ESTER VALUE

E-1 DEFINITION

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

Ethanol, 95 per cent (v/v).

Potassium hydroxide, approximately 0.5 N ethanolic solution. Prepare by dissolving 33 g of potassium hydroxide in 1 l of the ethanol. Allow to stand and decant or filter the clear liquid.

Potassium hydroxide, 0.1 N ethanolic solution.

Hydrochloric acid, 0.5 N solution.

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

E-3 APPARATUS

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

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 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 phenolphathalein 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.

Ignore any reappearance of the pink colour on standing.

E-5 CALCULATION OF ESTER VALUE

$$\text{Ester value } E = \frac{28.05 \times (B-V)}{M}$$

where

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

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

and B = mass, in grammes, of oil taken.

BUREAU OF CEYLON STANDARDS

The Bureau of Ceylon Standards (BCS) is the national standards organisation of Sri Lanka and was established by the Hon. Minister of Industries & Fisheries, as provided by the Bureau of Ceylon Standards Act, No. 38 of 1964.

The principal objects of the Bureau as set out in the Act are to promote standards in industry and commerce, prepare national standard specifications and codes of practice and operate a standardisation marks scheme and provide testing facilities, as the need arises.

The Bureau is financed by governmental grants and the sale of its publications. The financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The detailed preparation of standard specifications is done by Drafting Committees composed of experts in each particular field assisted by permanent officers of the Bureau. These committees are appointed by Divisional Committees, which are appointed by the Council. All members of the Drafting and Divisional Committees render their services in an honorary capacity. In preparing the standard specifications, the Bureau endeavours to ensure adequate representation of all view points.

In the international field the Bureau represents Sri Lanka in the International Organisation for Standardisation (ISO) and will participate in such fields of standardisation as are of special interest to Sri Lanka.

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