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SPECIFICATION FOR OIL OF CLOVE BUD

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BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR OIL OF CLOVE BUD

S. L. S. 247 : 1973

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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 CLOVE BUD

FOREWORD

This Sri Lanka Standard has been prepared by the Drafting Committee 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 5th December, 1973.

The oil of Clove bud is used extensively in the flavouring and cosmetic industries.

In the preparation of this standard the assistance derived from the publications of the British Standards Institution is acknowledged.

1. SCOPE

This Sri Lanka Standard defines certain characteristics of oil of clove bud with a view to facilitating assessment of its quality.

2. DEFINITION

The oil shall be the product obtained by steam distillation of the dried flower buds of *Eugenia caryophyllus* (Sprengel) Bullock and Harrison.

3. DESCRIPTION

The oil is colourless to yellow and has a characteristic odour of clove.

4. REQUIREMENTS

4.1 Odour and flavour – The assessment of odour and flavour shall be subject to agreement between the interested parties.

4.2 General appearance – The oil shall be clear, free from sediment, suspended matter, residual water and added adulterants.

4.3 Specific gravity at 30°C/30°C – The specific gravity of the oil at 30°C, when determined by the method described in Appendix A, shall be not less than 1.035 and not greater than 1.057.

4.4 Refractive index – The refractive index of the oil at 30°C, when determined by the method described in Appendix B, shall be not less than 1.5230 and not greater than 1.5310.

4.5 Solubility in ethanol – The solubility when tested by the method described in Appendix C, in 70% (v/v) ethanol at 30°C shall be 1 volume in 2 volumes.

4.6 Phenols – The phenolic content of the oil, when determined by the method described in Appendix D, shall be not less than 85% and not greater than 93% by volume.

4.7 Optical rotation – The optical rotation of the oil at 30°C, when determined by the method described in Appendix E, shall be within the range 0° to –15°.

5. PACKAGING AND MARKING

The oil shall be shipped preferably in glass, or tin lined containers, as agreed to between the purchaser and the supplier. A precipitate may result if galvanised iron containers are used. S.L.S. 212* and S.L.S. 211** are broadly applicable to Essential Oils.

6. STORAGE

The oil of clove should be stored in tight full containers protected from light.

7. 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, airtight, non-absorbent containers preferably glass 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. S.L.S. 213† and S.L.S. 210†† are broadly applicable to all essential oils.

APPENDIX A
SPECIFIC GRAVITY‡

A–1 The specific gravity of the material shall be expressed as the ratio of the mass in air of a given volume of the material at a specified temperature to that of an equal volume of water at the same temperature.

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* S.L.S. 212 – Sri Lanka Standard Method for Packaging of Essential Oils.

** S.L.S. 211 – Sri Lanka Standard Methods for Labelling and Marking of Containers for Essential Oils.

† S.L.S. 213 – Sri Lanka Standard Methods for the sampling of Essential Oils.

†† S.L.S. 210 – Sri Lanka Standard Method for the preparation of test sample of Essential Oils.

‡ The term adopted by the ISO is 'relative density' with reference to water.

A-1.1 The purchaser and the supplier may, by mutual agreement, fix any convenient temperature for the determination of specific gravity. A temperature of 30°C is recommended unless otherwise agreed to. Usually the specific gravity 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 specified in the individual standard or 0.000 64, if the correction factor is not so specified.

(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 specified in the individual standard, or 0.000 64, if the correction factor is not specified.

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

A-2 Specific gravity 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 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. Weigh accurately after standing for 30 minutes. Fill the clean, dried pyknometer or specific gravity bottle with the material 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 mass of the material contained in the pyknometer or specific gravity bottle divided by the water equivalent gives the specific gravity in air, of the material 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
REFRACTIVE INDEX

B-0 **GENERAL** – For the purpose of this determination, the refractive index of a 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 5 893 Å (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 (°C) at which the determination is made.

B-1 **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

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

the required temperature. Take a second reading after the lapse of a few minutes.

B-1.1 A temperature of 30°C is recommended. Carry out the determination at or as near as possible to the temperature specified.

B-1.2 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.

B-1.3 If, for any reason, the refractive index cannot be determined at the specified temperature, apply the correction factor specified in the individual standard or a factor of 0.000 38 per degree Celsius if a factor is not so specified. 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.

B-1.4 **Report** – Report the refractive index at 30°C as a number correct to four decimal places.

APPENDIX C
SOLUBILITY IN ALCOHOL

C-1 Unless otherwise specified, solubility in alcohol of the specified concentration shall be determined at 30°C. The temperature of the determination and the concentration of the alcohol used shall be reported.

C-2 PREPARATION OF SOLVENT

C-2.1 Solutions of alcohol in water that would be of the following percentage concentration (at the temperature of determination of the solubility) are used:

(a)	50	(c)	70	(e)	90
(b)	60	(d)	80	(f)	95

C-2.2 A convenient procedure for obtaining alcohol of the various concentrations given in Col. 1 of Table 1 is to weigh the alcohol (95 per cent by volume at the temperature of the

determination) and distilled water in the proportions by mass specified in Col. 2 and 3 and to mix them thoroughly. The strength of the alcohol at the temperature of determination shall be checked. Final adjustments shall be made, if necessary.

C-3 **APPARATUS** – A 10-ml glass-stoppered cylinder, graduated at 0.1 ml intervals.

C-4 **PROCEDURE** – Introduce exactly 1 ml of the liquid material into the cylinder and add, slowly and in small proportions, alcohol of the specified concentration, shaking the contents of the cylinder thoroughly after each addition. When a clear solution is first obtained, record the number of volumes of alcohol added. Continue the addition of alcohol until the 10-ml mark on the cylinder has been reached. If opalescence or cloudiness occurs during the subsequent additions of alcohol, note the point at which this phenomenon occurs. In case a clear solution is not obtained at any point during the addition of alcohol of one concentration, repeat the determination using alcohol of the next higher concentration.

C-5 **REPORT** – Report the behaviour of the material specifying the volume and the concentrations of the alcohol used corresponding to changes in the appearance of the mixture of the material and alcohol.

C-5.1 The terms clearly soluble, opalescent, and turbid, which are relative and entirely empirical, shall be used to describe the appearance of the solution in the following sense:

(a) Clearly soluble means that when the material and the solvent are mixed in the proportions stated, they form a clear and bright solution.

TABLE 1 – PREPARATION OF DILUTE ALCOHOLS

Alcohol (per cent by Volume) (1)	Proportions for mixing, by mass	
	Alcohol (95 per cent by Volume) (2)	Distilled Water (3)
50	460	540
60	564	436
70	676	324
80	796	204
90	927	73
95	1 000	0

(b) Opalescent means that the solution formed is not perfectly clear and bright, but is similar in appearance to solutions of standard opalescence prepared as described below:

Prepare three solutions by diluting 0.25 ml, 0.5 ml and 1.0 ml of 0.02 N sodium chloride solution to 50 ml with distilled water; then add 0.5 ml of 0.1 N silver nitrate solution, stir and view against a dark background, comparing the opalescence with that of the solution of the material through equal thickness of liquid. The resulting effects are:

(1) with 0.25 ml – faintly opalescent

(2) with 0.5 ml – slightly opalescent

(3) with 1.0 ml – distinctly opalescent

(c) Turbidity means that the solution formed is neither clear nor opalescent, but is similar in appearance to solutions of standard turbidity prepared as described below:

Prepare three solutions by diluting 0.25 ml, 0.5 ml and 1.0 ml of 0.1 N sulphuric acid to 50 ml with cold distilled water; then add 0.2 ml of approximately normal barium chloride solution; stir and allow to stand for 5 minutes at room temperature, and compare the turbidity with that of the solution of the material through equal thickness of liquid of the same type of glass container/test tube. The resulting effects are:

(1) with 0.25 ml – faintly turbid

(2) with 0.5 ml – slightly turbid

(3) with 1.0 ml – distinctly turbid

APPENDIX D

DETERMINATION OF PHENOLS

D–1 **REAGENTS** – The reagents used shall be of a recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

D–1.1 **Xylene** 3° – It should be tested to ensure freedom from impurities soluble in 5 per cent aqueous potassium hydroxide.

D-1.2 **Potassium hydroxide** – 5 per cent solution. Prepare by dissolving 59g of potassium hydroxide, in sufficient water to provide 1000 ml. Standardize to contain 5.0±0.1g of potassium hydroxide in 100 ml. The presence of more than traces of silica and alumina can give rise to separation of flocculent matter.

D-2 **APPARATUS** – A 150 ml flask.* Before use, thoroughly cleanse the flask with concentrated sulphuric acid and rinse out with distilled water.

D-3 **PROCEDURE** – Place in the flask 80 ml of the potassium hydroxide solution followed by 10 ml of the oil and shake the mixture thoroughly at five minute intervals during 30 minutes, at room temperature.

Raise the unabsorbed portion of the oil into the neck of the flask by the gradual addition of more of the potassium hydroxide solution, and facilitate the separation of the oily layer by rotating the flask between the hands and gently tapping. After leaving the flask to stand overnight, read off the volume of unabsorbed oil, taking the bottom line in each meniscus.

Where a small quantity, not exceeding 0.4 ml, of emulsion is formed between the oily and aqueous liquids, take a mean reading of this. If an emulsion is formed which will not separate, repeat the test with the addition of 2 ml of xylene to the test mixture before initial shaking. This facilitates the separation of the unabsorbed oil. Correct the final reading of unabsorbed oil for the added xylene.

D-4 **CALCULATION** – Phenols content of the oil, per cent by volume $= 10 \times V$ where V = volume in millilitres of oil absorbed.

Note 1 – Oil of Clove – In the case of this oil eugenol and aceto eugenol are both absorbed.

APPENDIX E
OPTICAL ROTATION

E-0 **GENERAL** – For the purpose of this determination, the optical rotation of a perfumery material is taken as the angle in degrees

* The flask is one of about 100–150 ml capacity with a narrow graduated neck. (Babcock type or any suitable modification).

through which the plane of polarization is turned when plane-polarized sodium light is passed through a layer of oil, 100 mm in thickness.

E-1 APPARATUS

E-1.1 **Polarimeter** – of suitable type with a precision of $\pm 0.03^{\circ}$. It should give a reading of 0° and also 180° with distilled water when properly adjusted.

E-1.2 **Light Source** – Any apparatus giving monochromatic light from sodium vapour lamp.

E-1.3 **Polarimeter tubes** – 100 ± 0.05 mm.

E-2 PROCEDURE

E-2.1 **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 20°C and the optical rotation should be $+ 34.62^{\circ}$.

E-2.2 Switch the light source on, and wait until full luminosity is obtained. Fill the polarimeter tube with the material at $30^{\circ} \pm 1^{\circ}$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.

E-2.3 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.

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