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SPECIFICATION FOR OIL OF GINGER

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BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR OIL OF GINGER

S.L.S. 215 : 1973

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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.

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SRI LANKA STANDARD SPECIFICATION FOR OIL OF GINGER

FOREWORD

This Sri Lanka Standard Specification 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 10th September 1973.

Oil of Ginger* is a volatile oil with the aromatic and persistent odour of ginger, but lacking the pungency or 'bite' usually associated with ginger. It is widely used as a flavour in soft drinks, baked products and in candies. In the preparation of this standard the assistance derived from publications of the Essential Oils Association of U.S.A. and publications of the British and Indian Standards Institutions is acknowledged.

1. SCOPE

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

2. DEFINITIONS

The oil shall be the product obtained by water or steam distillation of coarse, ground, rhizomes of *Zingiber officinale* Roscoe.

3. PHYSICAL REQUIREMENTS

3.1 **Solubility**—When determined at 30° C by the method described in Appendix A, the material shall be soluble with not more than a slight turbidity in one volume of absolute alcohol.

3.2 **Refractive Index**—The refractive index of the oil at 30° C determined by the method described in Appendix B shall be not less than 1.4840 and not more than 1.4894.

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*The Sinhala and Tamil common names for ginger are 'Inguru' and 'Inji', respectively.

3.3 **Specific Gravity**—The specific gravity of the oil at 30° C determined by the method described in Appendix C shall be not less than 0.868 and not more than 0.882.

3.4 **Optical Rotation**—Optical rotation of the oil at 30° C determined by the method described in Appendix D shall be within the range of –28° to –45°.

3.5 **Odour and Flavour**—The assessment of odour and flavour shall be subject to agreement between the interested parties.

4. CHEMICAL REQUIREMENTS

4.1 **Saponification Value**—The saponification value of the oil at 30° C on 5 g of the oil determined by the method described in Appendix E shall be less than 30.*

5. PACKAGING AND STORAGE† ††

5.1 **Containers**—The material shall be supplied in suitable containers as agreed to between the buyer and seller.

5.2 **Storage**—The material shall be stored preferably in tight full containers in a cool place protected from light.

6. SAMPLING

A representative sample or samples as required, each measuring not less than 25 ml shall be taken preferably from original containers from the bulk, for the purpose of examination. The sample shall be placed in clean, dry, dark glass bottles. The containers shall be of such size that they are nearly filled by the sample. Each container so filled shall be marked with the relevant details including date of sampling and shall be protected from light. The methods of sampling given in C.S. 213‡ are broadly applicable to oil of Ginger.

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*This value is tentative and is subject to review when further data become available.

†Refer C.S. 211 :—Methods for labelling and marking containers for Essential Oils.

††Refer C.S. 212 :—Methods for packing of Essential Oils.

‡ C.S. 213 :—Methods for sampling of Essential Oils.

APPENDIX A

DETERMINATION OF SOLUBILITY IN ALCOHOL

A-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.

A-2 Preparation of Solvent

A-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
(*b*) 60
(*c*) 70
(*d*) 80
(*e*) 90
(*f*) 95

A-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 percent 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.

A-3 Apparatus—A 10 ml glass stoppered cylinder, graduated at 0.1 ml intervals.

A-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.

TABLE 1.—PREPARATION OF DILUTE ALCOHOLS

Alcohol (Percent by Volume) (1)	Proportions for mixing by mass Alcohol (95 percent 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

A-5 Report—Report the behaviour of the material specifying the volume and the concentrations of the alcohol used corresponding to changes in the appearence of the mixture of the material and alcohol.

A-5.1 The terms clearly soluble, opalescent, and turbid, which are relative and entirely empirical, shall be used to describe the appearence 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.

(*b*) Opalescent means that the solution formed is not perfectly clear and bright, but is similar in appearence 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

(*e*) 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 B

DETERMINATION OF 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 A° (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 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.

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

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 nonobromnaphthalene, 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 reasons, 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

DETERMINATION OF *SPECIFIC GRAVITY (RELATIVE DENSITY)

C-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.

C-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:

---

*The term adopted by the International Organisation for Standardisation (ISO) is 'relative density' with reference to water. The ISO definition relates to the ratio of the true densities and not to the ratio of masses in air.

(*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 so specified.

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

C-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.

C-3 Procedure

C-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 of 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.

C-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.

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

APPENDIX D

DETERMINATION OF OPTICAL ROTATION

D-0 General—For the purpose of this determination, the optical rotation of a perfumery 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.

D-1 Apparatus

D-1.1 Polarimeter—Of suitable type with a precision of ± 0.03°. It should give a reading of 0° and also 180° with distilled water when properly adjusted.

D-1.2 Light Source—Any apparatus giving monochromatic light from sodium vapour lamp.

D-1.3 Polarimeter Tubes—100 ± 0.05 mm.

D-2 Procedure

D-2.1 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 20° C and the optical rotation should be + 34.62°.

D-2.2 Switch the light source on and wait until full luminosity is obtained. Fill the polarimeter tube with the material at 30° ± 1°C and ensure the absence of air bubbles. Place the tube in the polarimeter and read the dextro-rotatory (+) 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.

D-2.3 If the material is found to be excessively coloured, shake acid portion of the sample of the material with powdered tartaric/for about 5 minutes and filter. This treatment improves the colour of the material.

D-3 Specific Rotation

D-3.0 General—The optical activity is best determined in solution and the specific rotation is usually expressed by the symbol $(\alpha)^t_x$ in which t represents, in degrees centigrade, the temperature at which the rotation are is determined, and x represents the characteristic spectral line for wavelength of the light used. Spectral lines most frequently employed are the D line of sodium (doublet at 5 890 A° and 5 896 A°) and the yellow green line of mercury at 5 461 A°.

$$(\alpha)^t_x = \frac{100a}{l\,pd} = \frac{100a}{lc}$$

Where,

a = the corrected observed rotation in degrees of the solution at temperature t°, using D line of sodium or yellow-green line of mercury as the case may be;

l = the length of polarimeter tube in decimetres;

d = the specific gravity of the liquid or solution at the temperature of observations (t°);

p = the concentration of the solution expressed as the number of grammes of active substance in 100 g of solution; and

c = the concentration of solution expressed as the number of grammes of active substance in 100 ml of solution.

The symbol $(\alpha)_{x}^{t}$ is reserved exclusively for specific rotation; optical rotation determined in a 100-mm tube is indicated by α_{x}^{t} (the bracket being excluded).

D-3.1 Apparatus

D-3.1.1 Polarimeter—Generally, a polarimeter accurate to 0.05° of angular rotation, and capable of being read with the same precision, suffices. In the use of the polarimeter, the following general precautions shall be observed;

(*a*) The source of illumination shall be supplemented by a filtering system of transmitting light of a sufficiently monochromatic nature ;

(*b*) Precision polarimeters generally are designed to accommodate interchangeable discs to isolate the D line from sodium light or the 5 461 A° line from the mercury spectrum. With polarimeters not thus designed, cells containing suitably coloured liquids may be employed;

(*c*) Observations shall be accurate and reproducible to the extent that differences between replicates, or between observed and true values of rotation (the latter value having been established by calibration of the polarimeter scale with suitable standards), calculated in terms of specific rotation, shall not exceed one fourth of the range stipulated for the material;

(*d*) Polarimeter tubes shall be filled in such a way as to avoid creating or leaving air bubbles which interfere with the passage of the beam of light. Interference from bubbles is minimized with tubes in which the bore is expanded at one end;

(*e*) At the time of filling the tubes and the solution shall be at a temperature not higher than that specified for the determination, to guard against the formation of a bubble upon cooling and contraction of the contents; and

(*f*) In closing tubes consisting of removable end plates fitted with gaskets and caps, the latter shall be tightened only enough to ensure a leak-proof seal between the end plate and the body of the tube. Excessive pressure on the end plate may set up strains that result in interference with the measurement. In determining the specific rotation of a substance of low rotatory power, it is desirable to loosen the cap and tighten them again between successive readings in the measurement of both the rotation and the zero point. Any difference due to this factor will be revealed and appropriate adjustments to eliminate the cause may be made.

D-3.2 Reagents

D-3.2.1 Rectified Spirit

D-3.3 Procedure—Dissolve the material in rectified spirit reserving a separate portion of the solvent for a blank determination. Make at least five readings of the rotation of the solution at 30°C. Replace the solution with the reserved portion of the solvent, make the same number of readings and determine the average which shall be the zero point value. Subtract the zero point value from the average observed rotation, if the two figures are of the same sign, or add, if opposite in sign to obtain the corrected observed rotation.

D-3.4 Calculation—Calculate the specific rotation of the material using the formula given under D-3.0.

APPENDIX E

DETERMINATION OF SAPONIFICATION VALUE

E-0 General—Saponification value is equivalent to ester value plus acid value.

E-1 Acid Value

E-1.1 Reagents

E-1.1.1 Ethyl Alcohol—95 percent by volume, or rectified spirit. To remove impurities which might get coloured by potassium hydroxide solution and interfere with the sharpness of the end-point, reflux the 95 percent alcohol or rectified spirit over solid sodium hydroxide (NaOH) for 6 to 8 hours, and then distil.

E-1.1.2 Phenolphthalein Indicator—Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

E-1.1.3 Potassium Hydroxide Solution—0.1 N, approximately.

E-1.2 Procedure—Weigh accurately about 2.5 g of the dried material and dissolve in 20 ml of rectified spirit previously neutralized to phenolphthalein indicator with potassium hydroxide solution. Add about 10 drops of phenolphthalein indicator and titrate with potassium hydroxide solution until the solution remains faintly pink after 10 seconds of shaking.

E-1.3 Acid Value

$$\text{Acid value} = \frac{56.1\,vN}{M}$$

Where,

v = volume in ml of potassium hydroxide solution,

N = normality of the potassium hydroxide solution, and

M = mass in g of the material taken for the test.

Note: Sometimes certain samples of perfumery materials have very high acid values (20 and above). In such cases, 2.5 g of the material requires even more than 10 ml of 0.1 N aqueous potassium hydroxide solution. With highly acidic materials, it is recommended that 0.75 to 1 g of the material be taken instead of 2.5 g.

E-2 Ester Value

E-2.1 Reagents

E-2.1.1 **Ethyl Alcohol**—95 percent by volume or rectified spirit.

E-2.1.2 **Alcoholic Potassium Hydroxide Solution**—Approximately 0.5 N. Dissolve 33 g of potassium hydroxide in 1 000 ml of ethanol, 95 percent (v/v), allow to stand and decant or filter the clear liquid.

E-2.1.3 **Alcoholic Potassium Hydroxide Solution**—0.1 N.

E-2.1.4 **Hydrochloric Acid**—0.5 N, approximately.

E-2.1.5 **Phenolphthalein Indicator**—Dissolve 0.1 g of phenolphthalein in 60 percent rectified spirit.

E-2.2 Apparatus

E-2.2.1 **Flask**—250 ml capacity, made of chemically resistant glass and with a ground-glass interchangeable joint.

E-2.2.2 **Reflux Condenser**—with an interchangeable ground-glass joint.

E-2.3 **Procedure**—Weigh into the flask, to an accuracy of 1 mg, a suitable quantity of the material (see Table 2). Add 5 ml of ethyl alcohol or rectified spirit and neutralize with alcoholic potassium hydroxide solution using 2 ml of the phenolphthalein indicator. Add 25.0 ml of alcoholic potassium hydroxide solution and reflux the mixture for one hour. Cool and titrate the excess of alkali with hydrochloric acid using 0.5 ml of the phenolphthalein indicator. Carry out a blank determination following the same procedure but omitting the material.

Any reappearance of the pink colour on standing shall be ignored.

TABLE 2—MASS OF MATERIAL TO BE TAKEN FOR DETERMINATION OF ESTER VALUE

Expected Ester Value	Mass of the Material grammes
Below 50	4.5 to 5.0
50 to 70	3.5 to 4.0
70 to 90	2.5 to 3.0
90 to 110	2.0 to 2.5
110 to 140	1.5 to 2.0
140 to 180	1.2 to 1.5
180 to 220	1.0 to 1.2
220 to 228	0.9 to 1.0

E-2.4 Ester Value—Calculate the ester value of the material as follows:—

$$\text{Ester value} = \frac{(V_1 - V_2 \times N \times 56.1)}{M}$$

where,

V_1 = volume in ml of hydrochloric acid required for the blank,

V_2 = volume in ml of hydrochloric acid required to neutralize the excess alkali used for the hydrolysis,

N = normality of hydrochloric acid, and

M = mass in g of the material taken.

E-2.5 Calculation

Saponification value ester value + acid value.

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