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SPECIFICATION FOR CAMPHOR

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BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR CAMPHOR

S.L.S. 366 : 1975

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BUREAU OF CEYLON STANDARDS
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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 CAMPHOR

FOREWORD

This Sri Lanka Standard Specification has been prepared by the Drafting Committee of the Bureau on Camphor. 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 3rd December, 1975.

Camphor ($C_{10}H_{16}O$), is a naturally occurring bicyclic ketone, which is principally present in the volatile oils of camphor from "*Cinnamomum-camphora*" (Linn.). It is also obtained synthetically.

The standard values given in this specifications are in SI units.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final observed or calculated value shall be rounded off in accordance with C.S. 102: 1971* The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for camphor. This material is used in pharmaceutical preparations. It is also used as an incense in religious ceremonies.

2. TYPE

2.1 This standard covers two types of camphor, namely:

Type 1 — Pharmacopoeial, and
Type 2 — Technical

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* 102: 1971–Ceylon Standard on Presentation of Numerical Values.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall be either natural or synthetic, translucent or opaque and shall consist of crystals, granules or crystalline mass of tough consistency and pulverulent character, known as 'flowers of camphor': odour, penetrating and characteristic; taste, pungent, aromatic, and followed by a sensation of cold.

3.1.2 Type 2 material may also be in the form of powder or tablets.

3.2 **Identification**—The material shall burn readily with a bright, smoky, flame, and volatilise slowly at room temperature.

3.3 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in air-tight containers as agreed to between the purchaser and the supplier.

4.2 **Marking**—The following particulars shall be marked on each container.

(*a*) The word "camphor"
(*b*) The type
(*c*) The mass of the material
(*d*) The month and year of manufacture
(*e*) Batch number.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed in appropriate Appendices specified in Column 5 of Table 1.

6.2 **Quality of Reagents**—Unless otherwise specified, pure chemicals and distilled water shall be employed in tests.

TABLE 1—REQUIREMENTS FOR CAMPHOR

(See Clause 3.3)

Sl No.	Characteristic	Requirements for		Methods of Test in Appendix
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
(i)	Colour	Colourless or white, crystalline material	Colourless or white, crystalline material	—
(ii)	Solubility	Soluble in water ethyl alcohol, chloroform & ether under the conditions specified in Appendix A; freely soluble in fixed oils.	Soluble in water, ethyl alcohol, chloroform & ether under the conditions specified in Appendix A; freely soluble in fixed oils.	B
(iii)	Melting range°C	174 to 181	165 to 179	C
(iv)	Specific rotation (10 per cent (m/v) solution in 95 per cent alcohol)	Natural +40° to +43° and Synthetic -1.5° to +1.5°	Natural +40° to +43° and Synthetic -1.5° to +1.5°	D
(v)	Non-volatile matter, per cent by mass, max	0.1	0.1	E
(vi)	Total ketones, calculated as camphor ($C_{10}H_{16}O$) per cent by mass, min	96.0	90.0	F
(vii)	Freedom from halogens	To pass test	To pass test	G

APPENDIX A

SAMPLING PLAN FOR CAMPHOR

A-1 General Requirements of Sampling

A-1.1 General Precautions—In drawing, preparing, storing and handling samples, the following general precautions and directions shall be observed:

(*a*) Samples shall not be taken in an exposed place;

(*b*) The sampling implement and the containers for samples shall be made of aluminium or glass on which the material has no action. They shall be clean, dry and free from all odours;

(*c*) Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from contamination;

(*d*) To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both, by suitable means or by rolling;

(*e*) The sample containers shall be of such size that they are almost but not completely filled by the sample. The head space shall be between 5 to 10 per cent of the volume of the container depending on the method of transport adopted.

(*f*) Each sample container shall be sealed air-tight after filling and marked with full identification particulars, such as the sample number, the date of sampling, the month and year of manufacture of the material and any other relevant particulars of the consignment and

(*b*) Samples shall be stored in a cool place and protected from light and excessive variations of temperature.

A-1.2 Additional Precautions—The following additional precautions shall be observed:

(*a*) Rubber stoppers or composition corks shall not be used for closing the sample bottles;

(*b*) Samples shall be protected by covers or oil-proof plastics, aluminium or tin foil or any other suitable impervious material over the stopper to keep the moisture and dust away from the mouth of the bottle and to protect it while being handled and

(*c*) To avoid spoilage of the samples under no circumstances, shall sealing wax be applied direct to the cork.

A-2 Sampling Implements—A sampling scoop or any other suitable implement shall be used.

A-3 Scale of Sampling

A-3.1 Lot—In a single consignment of material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

A-3.2 For ascertaining the conformity of the lot to the requirements of material specifications, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

A-3.3 The containers shall be selected at random and to ensure randomness of selection, a random number table as agreed to between the purchaser and supplier shall be used. If necessary random number tables given in CS 77* : 1969, may be used. In case such a table is not available one of the following procedures is recommended for use.

(*a*) If the lot consists of individually packed containers then starting from any container, count them in any suitable order as 1, 2, 3.... up to r and so on where r is the integral part of N/n. Every rth container thus counted shall be withdrawn to give sample for tests.

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* C.S. 77 : 1969—Ceylon Standard Method of Sampling of Black Tea Packed in Containers.

TABLE 2—SCALE OF SAMPLING
(See Clause A-3.2)

Size of the Lot (N) (No. of Containers) (1)	Number of Containers to be selected (n) (2)
Up to 20	3
21 60	4
61 ,, 100	5
101 ,, 150	6
151 ,, 250	8
251 and above	10

NOTE:— In the case of very small lots where the selection of 3 containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be as agreed between the purchaser and the supplier.

(*b*) If the lot consists of cartons or cardboard boxes each containing more than one container, then a certain number of boxes (not less than 5 per cent of the total number of boxes in the lot wherever feasible) shall be chosen first. From each of the boxes so chosen, approximately equal number of containers shall then be selected so as to obtain the required number of containers specified in Table 2.

A-4 Preparation of Test Samples

A-4.1 From each of the containers selected according to Clause A-3.3, representative samples of the material, each sample containing not less than 200g shall be drawn with the help of the sampling implement.

A-4.2 Composite Sample—Out of these samples a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out determinations for all the characteristics specified under Clause A-5.2.

A-4.3 Individual Sample—The remaining portion of the material from each container shall form an individual sample.

A-4.4 The individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in Clause A-1.1 (f).

A-5 Number of Tests

A-5.1 Tests for the determination of melting range and total ketones shall be conducted on each of the individual samples.

A-5.2 Tests for the determination of all other characteristics given in clauses 3.1, 3.2, 3.3 and Table 1, shall be conducted on the composite sample.

A-6 Criteria for Conformity

A-6.1 For Individual Samples—A lot shall be declared as conforming to the requirements of melting range and total ketones, if each of the individual test results satisfies the relevant requirements given in Table 1.

A-6.2 For Composite Samples—For declaring conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results on each of the characteristics shall satisfy the relevant requirements given in clauses 3.1, 3.2, 3.3 and Table 1.

APPENDIX B

DETERMINATION OF SOLUBILITY
(See Table 1 Item ii)

B-1 Procedure

B-1.1 Add 800 ml of water to 1g of the material and stir for 3 hours at room temperature.

B-1.2 Add separately 1 ml of 95 per cent ethyl alcohol, 0.5 of ml of chloroform and 1 ml of ether to 1 g of the material at 27 ± 2°C.

APPENDIX C

DETERMINATION OF MELTING RANGE
(See Table 1, Item iii)

C-1 Principle—The melting range of a material is the range between the corrected temperature at which the material begins to form droplets and the corrected temperature at which it completely melts, as shown by the formation of a meniscus.

C-2 Apparatus

C-2.1 Capillary Tube—of soft glass, closed at one end, thickness of the wall about 0.10 mm to 0.15 mm; length, suitable for the apparatus used and of 0.9 mm to 1.1 mm internal diameter.

C-2.2 Thermometer—reading to an accuracy of 0.5 deg.

C-3 Procedure

C-3.1 Crush the sample to a fine powder with a clean stainless steel or horn spatula. Transfer some of it to a melting range tube of the size specified above. Pack the sample by tapping the end of the tube until the sample appears to be tightly packed, and essentially free from air space. Fill the tube to a depth of 3 mm to 4 mm in height. Attach the capillary tube and its contents to the thermometer so that the closed end is at a level at the middle of the bulb and heat in the heating vessel regulating the rise of temperature to within 25°C of the expected melting point. After that, regulate the rise of temperature to 3°C per minute until the first shrinkage in sample appears. Then regulate the heating in such a way that the rise during the melting interval is 0.5° per minute.

C-3.2 Note the temperature reading at which the material forms droplets against the side of the tube, and the temperature at which it is completely melted, as indicated by the formation of a definite meniscus.

APPENDIX D

DETERMINATION OF SPECIFIC ROTATION

(See Table 1 Item iv)

D-1 Principle—The optical activity of a solid is best determined in solution and the specific rotation is usually expressed by the symbol $(\alpha)_x^t$ in which t represents, in degrees centigrade, the temperature at which the rotation is determined, and α represents the characteristic spectral line for wave length of the light used. Spectral lines most frequently employed are the D line of sodium (doublet at 589.0 nm and 589.6 nm) and the yellow-green line of mercury at 546.1 nm.

$$\alpha \frac{t}{x} = \frac{100a}{lpd} = \frac{100a}{lc}$$

where

α = 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;

a = observed rotation;

l = the length of polarimeter tube in decimetres;

d = the relative density of the liquid or solution at the temperature of observation (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-2 Apparatus

D-2.1 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 capable 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 546.1 nm 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.

(*f*) In closing tubes consisting of removable end-plate 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 caps and tighten them again between successive readings in the measurement of both the rotation and the zero point. Any differences due to this factor will be revealed and appropriate adjustments to eliminate the cause may be made.

D-3 Procedure

D-3.1 Dissolve the material in ethyl alcohol (95% v.v) 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-4 Calculation

D-4.1 Calculate the specific rotation of the material using the formula given under Clause D—1.

APPENDIX—E

DETERMINATION OF NON-VOLATILE MATTER (See Table 1, Item v)

E-1 Principle—The sample is heated on a water-bath till most of the material has volatilized. The residue is heated to constant mass and determined as non-volatile matter content.

E-2 Procedure

E-2.1 Take 5 g of the sample in a tared platinum or silica dish and place it on a water-bath. Keep it heated till most of the material has volatilized. Transfer the dish to a hot air-oven maintained at 105 ± 1°C and heat to a constant mass. Cool in a desiccator and weigh.

E-3 Calculation

E-3.1 Non-volatile matter, per cent by mass $= \frac{m_1}{m_0} \times 100$

where,

m_1 = mass in g, of the residue, and

m_0 = mass in g, of the material taken for the test.

APPENDIX—F

DETERMINATION OF TOTAL KETONES (See Table 1 Item vi)

F-1 Principle—The ketonic constituents are converted into a 2:4 dinitrophenyl hydrazine derivative which is filtered out, washed, dried and weighed. Knowing the mass of the derivative, estimation of ketones as camphor can be carried out.

F-2 Reagents

F-2.1 Ethyl Alcohol (Aldehyde-Free)—95 per cent by volume. Reflux the alcohol gently over sodium hydroxide 3.5 per cent (m/v) together with aluminium shavings (1 per cent) for 8 to 10 hours. Distil suitably and collect the middle fraction. Keep the alcohol in an air-tight amber-coloured bottle.

F-2.2 2:4 Dinitrophenylhydrazine Solution—Dissolve 1.5 g of 2:4 dinitrophenylhydrazine in 20 ml of sulphuric acid (50 per cent v/v) dilute to 100 ml with water and filter. Solution shall be freshly prepared.

F-2.3 Dilute Sulphuric Acid—2 per cent (v/v), approximately.

F-3 Procedure

F-3.1 Dissolve about 0.2 g of the sample in 25 ml of aldehyde free ethyl alcohol, in a round-bottomed flask. To this, slowly with constant shaking, add 75 ml of 2:4 dinitrophenylhydrazine solution. Heat on a water bath for 4 hours under a reflux condenser. Remove the alcohol by distillation, allow to cool, dilute to 200 ml with dilute sulphuric acid and set aside for 24 hours. Filter the precipitate in a tared gooch crucible and wash with successive quantities of 10 ml of cold water until the washings are neutral to litmus paper. Dry at 80°C and weigh.

F-4 Calculation

F-4.1 Calculate using the following factor:

Each gramme of the precipitate is equivalent to 0.458 g of ketone (calculated as camphor, $C_{10}H_{16}O$).

APPENDIX—G

TEST FOR FREEDOM FROM HALOGENS

(See Table 1 Item vii)

G-1 Principle—The material is fused with sodium peroxide. The residue is dissolved in nitric acid and treated with standard silver nitrate solution. The turbidity produced is compared with a standard blank in which a definite quantity of hydrochloric acid is precipitated with silver nitrate solution.

G-2 Procedure

G-2.1 Mix 100 mg of the material with 200 mg of sodium peroxide in a clean, dry, hard glass test-tube. Suspend the tube at an angle of about 45° by means a of a clamp placed at the upper end, and gently heat the tube, starting at the upper end and gradually bringing the heat towards the lower part of the tube until fusion is complete. Dissolve the residue in 25 ml of warm water, acidify with dilute nitric acid and filter the solution into a Nessler tube. Wash the test tube and filter using two portions of 10 ml each of hot water, adding the washings to the filterered solution. To the filterate add 0.5 ml of 0.1 N silver nitrate solution, dilute with water to 50 ml, mix thoroughly and compare the turbidity with the control tube.

G-2.1.1 The material shall be deemed to have passed the test if the turbidity produced in the test is not greater than that produced in a control test with the same quantities of the reagents and 0.05 ml of 0.02 N hydrochloric acid.

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