SRI LANKA STANDARD 1016 : 1994

UDC 662 . 749.3 : 674.048

SPECIFICATION FOR COAL TAR CREOSOTE FOR USE IN TIMBER PRESERVATION

SRI LANKA STANDARDS INSTITUTION

SPECIFICATION FOR COAL TAR CREOSOTE FOR USE IN TIMBER PRESERVATION

SLS 1016 : 1994

Gr 16

SRI LANKA STANDARDS INSTITUTION
53, Dharmapala Mawatha,
Colombo 3,
Sri Lanka.

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.

SRI LANKA STANDARD
SPECIFICATION FOR COAL TAR CREOSOTE FOR USE IN TIMBER PRESERVATION

FOREWORD

This standard was approved by the Sectoral Committee on Chemicals and Chemical Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1994-03-31.

Coal tar creosotes consist essentially of mixtures of distillate oils from coal tar. Creosotes are resistant to leaching and are particularly suitable for exterior work. They can be used for interior work provided their odour is not objectionable. Creosotes have a characteristic odour which can be picked by foodstuffs and other materials in the vicinity without actually coming into contact with the treated wood. Creosotes have a certain degree of water repellency and can retard the movement of timber and the incidence of splitting, checking and distortion which tend to occur in timber exposed to the weather. They are not corrosive to metals and can have a protective action on iron and steel. Creosotes are not readily flammable and, after a few months of drying or weathering, creosoted wood presents no greater fire hazard than ordinary timber. Creosoted timber can be glued satisfactorily provided that the surface of the wood is free from surplus creosote. Porous materials such as plaster, if in contact with treated timber, tend to extract creosote by capillary action and become stained.

Guidelines for the determination of a compliance of a lot with the requirements of this standard based on statistical sampling and inspection are given in Appendix A.

This preservative contains substances which are injurious to health if adequate precautions are not taken. It is important to wear, wherever necessary, protective clothing such as rubber or plastic coated gloves, face masks, goggles and head and foot gears. The hazards from the preservative should be well understood by all the staff and recommended code of practice should be followed. In case of an accident first-aid treatment to be followed is given in Appendix N as a guidance.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this standard, the assistance derived from the following publications is gratefully acknowledged :

BS 144 : 1990 Wood preservation using coal tar creosotes
Part 1 : Specification for preservative.
BS 1282 : 1975 Guide to the choice, use and application of wood preservatives.
IS 218 : 1983 Creosote oil for use as wood preservatives.

1 SCOPE

This specification prescribes the requirements and methods of test for coal tar creosote for use in timber preservation.

2 REFERENCES

CS 102 Presentation of numerical values.
CS 124 Test sieves.
SLS 428 Random sampling methods.
SLS 584 Methods of test for petroleum and petroleum products.
SLS 692 Safety colours and safety signs.

3 TYPES

The following types of coal tar creosote for use in timber preservation are covered in this specification :

Type 1 - intended for treatment of timber by pressure impregnation.
Type 2 - intended for treatment by pressure impregnation but has a more closely defined distillation range and a more restricted residue content than Type 1. It is especially suitable for treatment of poles for overhead power and telecommunication lines and for structural timbers where bleeding in service could present a problem.
Type 3 - intended for the treatment of wood by immersion, spraying or brushing.

NOTE

All these three types may also be used for the hot-and-cold open tank process.

4 REQUIREMENTS

4.1 Composition

All types of creosotes shall consist of blends of distillates of coal tar and shall be free from petroleum oils or oils not derived from coal tar.

4.2 Liquidity

4.2.1 The creosotes of Type 1 and Type 2 when tested by the method prescribed in Appendix C shall be free from any solid matter until the end of 2 hours at 32 °C.

4.2.2 The creosotes of Type 3 when tested by the method prescribed in Appendix C shall be free from any solid matter until the end of 4 hours at 0 °C.

4.3 Other requirements

Creosotes shall also comply with the requirements given in Table 1, when tested by the methods given in Column 6 of the table.

5 PACKAGING AND MARKING

The product shall be suitably packed. Each container shall be legibly and indelibly marked or labelled with the following :

a) Name of the product;
b) Type;
c) Pictorial marking for toxic nature with colours and dimensions as given in SLS 692 (see Figure 1);
d) Net volume, in litres;
e) Name and address of the manufacturer and/or supplier (including the country of origin) ; and
f) Batch identification mark.

FIGURE 1 - Safety sign to indicate toxic nature

NOTE

Attention is drawn to the certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of this specification.

TABLE 1 - Requirements for coal tar creosotes for timber preservation

Sl. No. (1)	Characteristic (2)	Requirements for			Method of test (6)
		Type 1 (3)	Type 2 (4)	Type 3 (5)	
i)	Density, kg/m³, at				Appendix D
	a) 38 °C	1003-1008	1003-1008	-	
	b) 20 °C	-	-	910-1120	
ii)	Distillation characteristics, recovery of dehydrated creosote, per cent by mass, at				Appendix E
	a) 205 °C	6 max	5 max	35 max	
	b) 230 °C	40 max	5-30	55 max	
	c) 270 °C	-	-	20 min	
	d) 315 °C	78 max	40-78	40-85	
	e) 355 °C	60 min	79-30	65 min	
iii)	Extractable phenols content in the distillate up to 315 °C (as obtained in Appendix E), ml/100 g of dehydrated creosote				Appendix F
	a) Type 1 and Type 2 density range 1003 kg/m³ to 1045 kg/m³	5-20	5-20	-	
	b) Type 1 and Type 2, density range 1046 kg/m³ to 1108 kg/m³	0-20	0-20	-	
	c) Type 3	-	-	1-20	

Table 1 - (concluded)

Sl. No.	Characteristic	Requirements for Type 1	Type 2	Type 3	Method of test
iv)	Extractable phenols content boiling upto 220 °C, ml/100 g of dehydrated creosote, max.	-	-	4	Appendix G
v)	Flash point, Pensky Martens closed tester, °C, min.	66	66	66	M 6 of SLS 584 Volume 1
vi)	Kinematic viscosity at 40 °C, mm^2/s	-	4-14		Appendix H
vii)	Water content, per cent by volume, max.	1.5	1.5	1.5	Appendix J
viii)	Insoluble matter content, per cent by mass, max.	0.4	0.4	0.4	Appendix K
ix)	Saturated hydrocarbon content per cent by mass, max.	15	15	25	Appendix L
x)	Napthalene content, per cent by mass	-	8-25	-	Appendix M

6 METHODS OF TEST

6.1 Tests shall be carried out as prescribed in Method M 5 of SLS 584 Volume 1 : 1982 and Appendices B to M of this specification.

6.2 Unless otherwise specified reagents of analytical grade and distilled water or water of equivalent purity shall be used.

APPENDIX A
COMPLIANCE OF A LOT

The sampling scheme given in this Appendix should be applied where compliance of a lot to the requirements of this standard is to be assessed based on statistical sampling and inspection. Where compliance with this standard is to be assured based on manufacturer's control systems coupled with type testing and check tests or any other procedure, appropriate scheme of sampling and inspection should be adopted.

A.1 LOT

All containers of coal tar creosotes of one type and belonging to one batch of manufacture or supply shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

A.2.1 When drawing and handling samples of coal tar creosotes, necessary safety precautions shall be observed.

A.2.2 Samples shall be placed in clean air-tight glass containers or cans but shall not be closed with rubber stoppers.

A.3 SCALE OF SAMPLING

A.3.1 The number of containers to be selected from a lot should be in accordance with Table 2.

TABLE 2 - Scale of sampling

Number of containers in a lot	Number of containers to be selected
Up to 15	2
16 to 50	3
51 to 150	5
151 to 500	8

A.3.2 The containers shall be selected at random. In order to ensure randomness of selection tables of random numbers as given in SLS 428 shall be used.

A.4 PREPARATION OF COMPOSITE SAMPLE

A.4.1 A sufficient quantity of material shall be drawn from top, middle, and bottom portions of each container selected as in A.3.1 with the help of a sampling tube.

A.4.2 The material drawn from all the containers shall be thoroughly mixed together and transferred to a sample container. This container shall be sealed and marked with necessary details of sampling.

A.5 NUMBER OF TESTS

A.5.1 Each container selected as in A.3.1 shall be inspected for packaging and marking requirements.

A.5.2 The composite sample prepared as in A.4 shall be tested for the requirements given in 4.2 and 4.3.

A.6 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied:

A.6.1 Each container inspected as in A.5.1 satisfies the packaging and marking requirements.

A.6.2 The test results of the composite sample, tested as in A.5.2 satisfy the relevant requirements.

APPENDIX B
PREPARATION OF LABORATORY SAMPLE

PROCEDURE

Place the sample in a clean, dry, air-tight container which may be a glass bottle or a can, but which is not closed with a rubber stopper. Allow an ullage of about 5 per cent in the sample container.

Examine the sample for the presence of free water and crystalline solids and report the presence or absence of these.

Mix the sample thoroughly immediately before any portion is withdrawn. If it shows signs of containing precipitated solids, immerse the container in a water-bath at a temperature just high enough to dissolve these solids, and take portions for test from the warmed, mixed sample.

APPENDIX C
DETERMINATION OF LIQUIDITY

C.1 APPARATUS

C.1.1 **Conical flask,** 100-ml capacity.

C.2.2 **Thermometer,** a partial immersion thermometer of range -0.5 °C to 40.5 °C graduated at each 0.1 °C and accurate to ± 0.2 °C.

C.2.3 **Constant temperature baths,** depending on the type of creosote being tested, baths capable of maintaining temperature of 38 ± 0.1 °C, 32 ± 0.1 °C, 10 ± 0.1 °C or 0 ± 0.1 °C.

C.2 PROCEDURE

Pour about 50 ml of the sample (see Appendix B) into the conical flask. Fit the thermometer by means of a cork into the neck of the flask, with the bulb of the thermometer immersed in the creosote. Place the flask in the appropriate constant temperature bath, i.e. 38 ± 0.1 °C for Type 1 and Type 2 and 10 ± 0.1 °C for Type 3.

Ensure that the surface of the creosote is below that of the water in the bath. Swirl the flask until the creosote reaches the appropriate temeperature. Leave the flask in the bath for 2 hours, then withdraw the flask and examine the surface of the creosote for the presence of solid matter. Rotate the flask slowly with the side of the flask in a horizontal position and examine the inside surface of the flask for the presence of solid matter.

When the sample is free from solid matter, place the flask in the second bath maintained at 32 ± 0.1 °C for Type 1 and Type 2 creosote and 0 ± 0.1 °C for Type 3 creosote.

Re-examine the sample for solids, as before, when the contents have reached the bath temperature and again after having maintained the sample at that temperature for 2 hours for Type 1 and Type 2 or, in the case of Type 3, after 4 hours.

C.3 EXPRESSION OF RESULTS

The sample is deemed to pass the liquidity test if it remains completely liquid for the specified period at the specified temperature.

The sample is deemed to fail the liquidity test if any solid matter is observed either after cooling to the specified temperature or at any time up to the end of the specified period at that temperature.

APPENDIX D
DETERMINATION OF DENSITY

D.1 APPARATUS

D.1.1 Hydrometer vessel, a cylindrical glass vessel, free from local irregularities producing distortion and several millimeters greater in diameter than that of the hydrometer bulb.

D.1.2 Hydrometer, a density hydrometer, capable of measuring density ranges given in Table 1.

NOTE

Hydrometers calibrated in g/ml or kg/m³ may be used. If a hydrometer calibrated in kg/m³ is used, read it to the nearest kg/m³ (see D.2), multiply the corrections described in D.3.1 by 1 000, and omit the factor 1 000 from the equation in D.3.2.

Examine the hydrometer before use to see that it is clean and dry and that there has been no displacement of the paper scale during use. Any displacement of the paper can be detected by reference to the means provided for this purpose, for example, a horizontal line etched on the stem and the corresponding datum marked on the paper scale, thus >---<. If the scale has been displaced, recertification of the hydrometer is necessary.

D.1.3 Thermometer, , a partial immersion thermometer of range -0.5 °C to 40.5 °C, graduated in 0.1 °C and accurate to ± 0.2 °C.

D.2 PROCEDURE

Bring the sample (see Appendix B) to approximately 38 °C for Type 1 and Type 2, and to approximately 20 °C for Type 3. Fill the clean hydrometer vessel (D.1.1) with the liquefied sample (see Note) to a sufficient depth, so that the hydrometer will not touch the bottom of the vessel when it is completely immersed in the sample. To avoid the formation of air bubbles pour the sample down the side of the vessel. Stir the sample, again avoiding the formation of air bubbles. Hold the hydrometer (D.1.2) by the top of the stem, insert it carefully into the sample and release it when approximately in the position of equilibrium, so that the hydrometer rises or falls only by a small amount.

NOTE

Immediately before drawing samples, mix the sample thoroughly. If it shows signs of containing precipitated solids, immerse the container in a water bath at a temperature just high enough to dissolve these solids and take portions for test from the warmed, mixed sample.

Lightly press down the top of the hydrometer stem so that the stem is immersed a few millimetres more. Release the hydrometer. After a few oscillations, note the reading when the hydrometer is steady.

Observe the meniscus during this period. If the stem is clean the meniscus shape will remain unchanged during the hydrometer oscillations; if the meniscus shape changes, clean the hydrometer and repeat the procedure.

Record the hydrometer reading to the nearest 0.001 g/ml and also the temperature of the creosote to the nearest 0.1 °C. Take the hydrometer reading for opaque creosotes at the level where the meniscus merges into the stem of the hydrometer.

D.3 CALCULATION

D.3.1 Correction of hydrometer reading

Corrected hydrometer reading $R_T = R + C + 0.0007$

where,

R is the hydrometer readings in g/ml;
C is the certification correction; and
0.0007 is the meniscus height correction to be used for opaque creosotes only.

D.3.2 Calcualtion of density

Density at the required temperature,
(20 °C or 38 °C), $kg/m^3 = 1\ 000\ (R_T m + a)$

where,

R_T is the corrected hydrometer reading, in g/ml (see D.3.1);
m is the multiplication factor (see Note);and
a is the addition factor (see Note).

NOTE

m and a are obtained from Table 3 for Type 1 and Type 2, and from Table 4 for Type 3.

TABLE 3 - Multiplication (m) and addition (a) factors for the conversion of corrected hydrometer reading at t°C to density at 38 °C (for Types 1 and Type 2)

t° C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
34	1.003 18	1.003 09	1.002 99	1.002 90	1.002 81	1.002 71	1.002 62	1.002 53	1.002 43	1.002 34
	-0.006 67	-0.006 50	-0.006 34	-0.006 17	-0.006 00	-0.005 84	-0.005 67	-0.005 50	-0.005 33	-0.005 17
35	1.002 25	1.002 15	1.002 06	1.001 97	1.001 88	1.001 78	1.001 69	1.001 60	1.001 50	1.001 41
	-0.005 00	-0.004 83	-0.004 66	-0.004 50	-0.004 33	-0.004 16	-0.004 00	-0.003 83	-0.003 66	-0.003 50
36	1.001 32	1.001 22	1.001 13	1.001 04	1.000 94	1.000 85	1.000 76	1.000 67	1.000 57	1.000 48
	-0.003 33	-0.003 16	-0.003 00	-0.002 83	-0.002 66	-0.002 50	-0.002 33	-0.002 16	-0.002 00	-0.001 83
37	1.000 39	1.000 30	1.000 20	1.000 11	1.000 02	0.999 92	0.999 83	0.999 74	0.999 64	0.999 55
	-0.001 66	-0.001 50	-0.001 33	-0.001 16	-0.001 00	-0.000 83	-0.000 67	-0.000 50	-0.000 33	-0.000 17
38	0.999 46	0.999 37	0.999 28	0.999 18	0.999 09	0.999 00	0.998 90	0.998 81	0.998 72	0.998 63
	0.000 00	0.000 17	0.000 33	0.000 50	0.000 66	0.000 83	0.001 00	0.001 16	0.001 33	0.001 49
39	0.998 53	0.998 44	0.998 35	0.998 26	0.998 16	0.998 07	0.997 98	0.997 89	0.997 80	0.997 70
	0.001 66	0.001 83	0.001 99	0.002 16	0.002 32	0.002 49	0.002 66	0.002 82	0.002 99	0.003 15
40	0.997 61	0.997 52	0.997 43	0.997 33	0.997 24	0.997 15	0.997 06	0.996 97	0.996 87	0.996 78
	0.003 32	0.003 48	0.003 65	0.003 81	0.003 98	0.004 14	0.004 31	0.004 48	0.004 64	0.004 81
41	0.996 69	0.996 60	0.996 50	0.996 41	0.996 32	0.996 23	0.996 14	0.996 04	0.995 95	0.995 86
	0.004 97	0.005 13	0.005 30	0.005 47	0.005 63	0.005 80	0.005 96	0.006 13	0.006 29	0.006 46

NOTE : The upper figure is the multiplication factor.

TABLE 4 - Multiplication (m) and addition (a) factors for the conversion of corrected hydrometer reading at t°C to density at 20 °C (for Type 3)

t° C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
10	1.009 35 -0.016 77	1.009 26 -0.016 60	1.009 17 -0.016 43	1.009 07 -0.016 26	1.008 98 -0.016 09	1.008 88 -0.015 92	1.008 79 -0.015 75	1.008 69 -0.015 59	1.008 60 -0.015 42	1.008 51 -0.015 25
11	1.008 41 -0.015 08	1.008 32 -0.014 91	1.008 22 -0.014 74	1.008 13 -0.014 57	1.008 03 -0.014 40	1.007 94 -0.014 23	1.007 85 -0.014 07	1.007 75 -0.013 90	1.007 66 -0.013 73	1.007 56 -0.013 56
12	1.007 47 -0.013 39	1.007 38 -0.013 22	1.007 28 -0.013 05	1.007 19 -0.012 89	1.007 09 -0.012 72	1.007 00 -0.012 55	1.006 91 -0.012 38	1.006 81 -0.012 21	1.006 72 -0.012 04	1.006 62 -0.011 88
13	1.006 53 -0.011 71	1.006 44 -0.011 54	1.006 34 -0.011 37	1.006 25 -0.011 20	1.006 15 -0.011 03	1.006 06 -0.010 87	1.005 97 -0.010 70	1.005 87 -0.010 53	1.005 78 -0.010 36	1.005 69 -0.010 19
14	1.005 59 -0.010 03	1.005 50 -0.009 86	1.005 40 -0.009 69	1.005 31 -0.009 52	1.005 22 -0.009 35	1.005 12 -0.009 19	1.005 03 -0.009 02	1.004 94 -0.008 85	1.004 84 -0.008 68	1.004 75 -0.008 52
15	1.004 66 -0.008 35	1.004 56 -0.008 18	1.004 47 -0.008 01	1.004 38 -0.008 85	1.004 28 -0.007 68	1.004 19 -0.007 51	1.004 10 -0.007 34	1.004 00 -0.007 18	1.003 91 -0.007 01	1.003 82 -0.006 84
16	1.003 72 -0.006 67	1.003 63 -0.006 51	1.003 54 -0.006 34	1.003 44 -0.006 17	1.003 35 -0.006 00	1.003 26 -0.005 84	1.003 16 -0.005 67	1.003 07 0.005 50	1.002 98 -0.005 33	1.002 88 -0.005 17
17	1.002 79 -0.005 00	1.002 70 -0.004 83	1.002 60 -0.004 67	1.002 51 -0.004 50	1.002 42 -0.004 33	1.002 32 -0.004 16	1.002 23 -0.004 00	1.002 14 -0.003 83	1.002 04 -0.003 66	1.001 95 -0.003 50
18	1.001 86 -0.003 33	1.001 76 -0.003 16	1.001 67 -0.003 00	1.001 58 -0.002 83	1.001 49 -0.002 66	1.001 39 -0.002 50	1.001 30 -0.002 33	1.001 21 -0.002 16	1.001 11 -0.002 00	1.001 02 -0.001 83
19	1.000 93 -0.001 66	1.000 84 -0.001 50	1.000 74 -0.001 33	1.000 65 -0.001 17	1.000 56 -0.001 00	1.000 46 -0.000 83	1.000 37 -0.000 67	1.000 28 -0.000 50	1.000 19 -0.000 33	1.000 09 -0.000 17
20	1.000 00 0.000 00	0.999 91 0.000 17	0.999 82 0.000 33	0.999 72 0.000 50	0.999 63 0.000 67	0.999 54 0.000 83	0.999 44 0.001 00	0.999 35 0.001 17	0.999 26 0.001 33	0.999 17 0.001 50

NOTE : The upper figure is the multiplication factor.

TABLE 4 - (concluded)

t° C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
21	0.999 07	0.998 98	0.998 89	0.998 80	0.998 70	0.998 61	0.998 52	0.998 43	0.998 33	0.998 24
	0.001 66	0.001 83	0.002 00	0.002 16	0.002 33	0.002 50	0.002 66	0.002 83	0.003 00	0.003 16
22	0.998 15	0.998 06	0.997 97	0.997 87	0.997 78	0.997 69	0.997 60	0.997 50	0.997 41	0.997 32
	0.003 33	0.003 50	0.003 66	0.003 83	0.004 00	0.004 16	0.004 33	0.004 50	0.004 67	0.004 83
23	0.997 23	0.997 13	0.997 04	0.996 95	0.996 86	0.996 77	0.996 67	0.996 58	0.996 49	0.996 40
	0.005 00	0.005 17	0.005 33	0.005 50	0.005 67	0.005 84	0.006 00	0.006 17	0.006 34	0.006 51
24	0.996 31	0.996 21	0.996 12	0.996 03	0.995 94	0.995 85	0.995 75	0.995 66	0.995 57	0.995 48
	0.006 67	0.006 84	0.007 01	0.007 18	0.007 34	0.007 51	0.007 68	0.007 85	0.008 01	0.008 18
25	0.995 39	0.995 29	0.995 20	0.995 11	0.995 02	0.994 93	0.994 84	0.994 74	0.994 65	0.994 56
	0.008 35	0.008 52	0.008 68	0.008 85	0.009 02	0.009 19	0.009 35	0.009 52	0.009 69	0.009 86
26	0.994 47	0.994 38	0.994 29	0.994 19	0.994 10	0.994 01	0.993 92	0.993 83	0.993 73	0.993 64
	0.010 03	0.010 19	0.010 36	0.010 53	0.010 70	0.010 87	0.011 03	0.011 20	0.011 37	0.011 54
27	0.993 55	0.993 46	0.993 37	0.993 28	0.993 19	0.993 09	0.993 00	0.992 91	0.992 82	0.992 73
	0.011 71	0.011 88	0.012 04	0.012 21	0.012 38	0.012 55	0.012 72	0.012 89	0.013 05	0.013 22
28	0.992 64	0.992 55	0.992 45	0.992 36	0.992 27	0.992 18	0.992 09	0.992 00	0.991 91	0.991 82
	0.013 39	0.013 56	0.013 73	0.013 90	0.014 07	0.014 23	0.014 40	0.014 57	0.014 74	0.014 91
29	0.991 72	0.991 63	0.991 54	0.991 45	0.991 36	0.991 27	0.991 18	0.991 09	0.991 00	0.990 90
	0.015 08	0.015 25	0.015 42	0.015 59	0.015 75	0.015 92	0.016 09	0.016 26	0.016 43	0.016 60

NOTE : The upper figure is the multiplication factor.

APPENDIX E
DETERMINATION OF DISTILLATION CHARACTERISTICS

E.1 APPARATUS

E.1.1 Distillation apparatus, as described in Method M 5 of SLS 584 : Volume 1 : 1982 except as otherwise stated in E.1.1.1 to E.1.1.4.

E.1.1.1 *Distillation flasks*

a) 250-ml capacity, untared;and
b) 150-ml capacity, tared; held in the vertical position by means of a clamp at the extreme upper end of the neck.

E.1.1.2 *Condensers*

a) fluid-cooled type as described in 4.2.1 of method M 5 of SLS 584 : Volume 1 : 1982;and
b) air-cooled type as given in Figure 2.

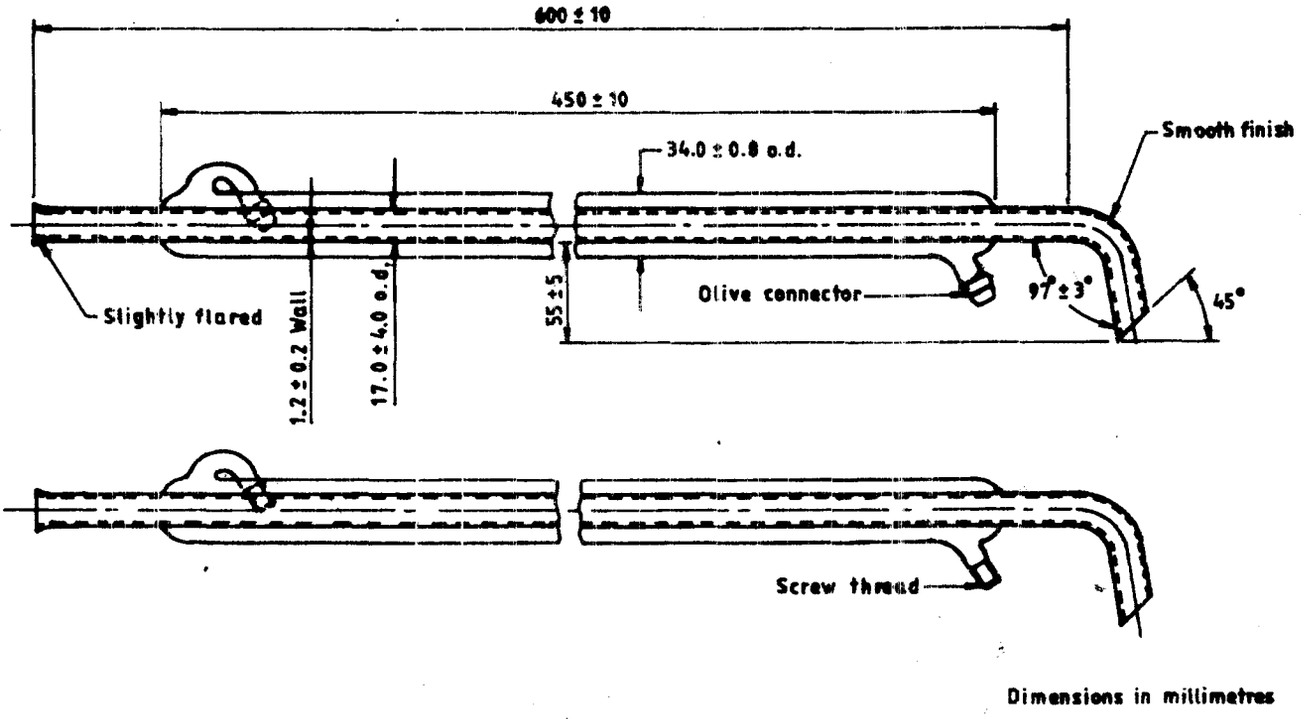

FIGURE 2 - Air-cooled type condenser

E.1.1.3 *Metal shield or enclosure for flask,* Type 1 as described in 4.3.1. of Method M 5 of SLS 584 : Volume 1 : 1982.

E.1.1.4 *Crow receivers,* 50-ml capacity, and tared.

E.1.2 **Thermometer,** having a range -2 °C to 400 °C.

E.1.3 **Separating funnels,** capacity 50-ml.

E.1.4 **Barometer.**

E.2 **PROCEDURE**

E.2.1 **Corrections**

E.2.1.1 *General*

Apply the corrections described in E.2.1.2 and E.2.1.3 to the specified distillation temperatures before commencing the distillation.

E.2.1.2 *Barometer readings*

E.2.1.2.1 Read the barometer to obtain the atmospheric pressure in mbar or mmHg and record the ambient temperature. Continue as described in either E.2.1.2.2 or E.2.1.2.3.

NOTE

If atmospheric pressure is measured in other units the following conversion factors may be used:

1 mbar = 100 N/m^2 = 100 Pa
1 mmHg = 133.322 N/m^2.

E.2.1.2.2 Correct the barometer readings for temperature. If the corrected reading differs from 1 013 mbar, apply corrections to the specified distillation temperature by adding the value given in Table 5 for every 1 mbar above 1 013 mbar or subtracting the value for every 1 mbar below 1 013 mbar.

E.2.1.2.3 Correct the barometer readings for temperature. If the reading differs from 760 mmHg, apply corrections to the specified distillation temperature by adding the value given in Table 5 for every 1 mmHg above 760 mmHg or subtracting the value for every 1 mmHg below 760 mmHg.

TABLE 5 -- Corrections for barometric pressure

Specified temperature	Corrections	
	per mbar	per mmHg
°C	°C	°C
205	0.076	0.057
230	0.080	0.060
270	0.087	0.065
315	0.095	0.071
355	0.100	0.075

E.2.1.3 *Thermometers*

If necessary, make the appropriate adjustments indicated by the thermometer test certificate, at any of the specified distillation temperatures (see Table 1).

E.2.2 Preparation of test portion of dehydrated creosote

Transfer about 120 g of the sample (see Appendix B) into the 250-ml untared distillation flask **(E.1.1.1.a)** to which has been added fragments of porous inert material. Using the fluid-cooled type condenser **(E.1.1.2.a)** distill the sample and collect the distillate in a separating funnel **(E.1.3)**, stopping the distillation when water ceases to distil. Allow the contents of the separating funnel to settle, run the lower water layer off, and return the oil layer to the distillation flask when the flask has cooled to about 40 °C. Mix the contents of the flask thoroughly, ensuring that the oil is homogeneous.

E.2.3 Distillation

Weigh, to the nearest 0.1 g, approximately 100 g of the dehydrated creosote **(E.2.2)**, directly into the tared 150-ml distillation flask **(E.1.1.1.b)**.

Add fragments of porous inert material and assemble the apparatus, with the side arm of the flask extending at least 25 mm beyond the cork in the upper end of the air-cooled type condenser **(E.1.1.2.b)**

Using the naked flame of a Bunsen burner, distil at a rate of 5 ± 0.5 ml/minute. If for any reason the distillation rate falls outside the specified limits at any time after the first 5 ml of distillate have been collected, discontinue the test and start another on a further portion of the original sample.

NOTE

The specified distillation rate corresponds to approximately 90 drops per minute, i.e.3 drops in 2 s but this figure should be taken only as a guide in order that the rate in millilitres per minute may be kept under close observation.

If solids are deposited in the condenser during the distillation, warm the condenser so that such solids are collected in the fraction with which they distil.

Change the receiver at each corrected specified temperature (see Tables 1 and 5), without stopping the distillation.

Extinguish the flame when the thermometer indicates the highest corrected specified temperature. The final fraction includes the oil which drains from the condenser within 5 minutes after the flame has been extinguished.

Weigh each receiver containing distillate fraction. Note the mass of each fraction (f_1, f_2, f_3, f_4 and f_5).

Reserve the distillate fractions f_1, f_2, f_3, and f_4 for test according to Appendix F and additionally Appendix G for Type 3 creosotes.

E.3 CALCULATION

Let,

f_1 be the mass of the fraction distilling up to 205 °C;
f_2 be the mass of the fraction distilling from 205 °C to 230 °C;
f_3 be the mass of the fraction distilling from 230 °C to 270 °C;
f_4 be the mass of the fraction distilling from 270 °C to 315 °C; and
f_5 be the mass of the fraction distilling from 315 °C to 355 °C.

$$\text{Recovery of dehydrated creosote at temperature } T_i, \text{ per cent by mass} = \frac{100 \sum_{i=1}^{n} f_i}{m}$$

where,

n = 1 to 5 as appropriate;
f_i is the mass, in g, of cumulative distillate, upto the temperature T_i; and
m is the mass, in g, of dehydrated creosote used for the test.

APPENDIX F
DETERMINATION OF EXTRACTABLE PHENOLS

F.1 REAGENTS

F.1.1 **Sodium hydroxide,** 100 g/l solution.

F.1.2 **Sodium chloride,** saturated solution.

F.1.3 **Methyl orange indicator,** 1g/l solution.

F.1.4 **Hydrochloric acid,** concentrated (rel. den.=1.18).

F.1.5 **Sodium chloride,** powdered solid.

F.2 APPARATUS AND MATERIALS

F.2.1 **Separating funnel,** capacity 250-ml, stoppered.

F.2.2 **Phenols flask,** 200-ml or 150-ml capacity.

F.2.3 **Glass wool.**

F.2.4 **Measuring cylinder,** 100-ml capacity.

F.2.5 **Thermometer,** range - 10 °C to 110 °C.

F.3 PROCEDURE

F.3.1 The test portion (see Note 1) shall be obtained by combining distillate fractions ($f_1+f_2+f_3+f_4$) collected below 315 °C as in Appendix E.

F.3.2 If necessary warm the test portion until completely liquid, and transfer it to the separating funnel (F.2.1). Use 50 ml of the sodium hydroxide solution (F.1.1) (see Notes 2 and 3) to rinse the receivers from which the test samples were transferred and add it to the separating funnel. If the combined samples contain solids which have separated, warm the separating funnel just sufficient to redissolve the solids.

Stopper the funnel; shake it vigorously for 1 minute to 2 minutes and allow it to stand. After separation (see Note 4) run the alkaline layer into a beaker, repeat this operation with successive 25-ml portions of the sodium hydroxide solution (F.1.1) until all the phenols have been removed from the oil layer (see Notes 2 and 3).

After separation, add the alkaline layers to the first sodium hydroxide washing. A large excess of sodium hydroxide has to be avoided but some excess has to be maintained.

During the extraction procedure ensure that the contents of the separating funnel are completely liquid by immersing the funnel, if necessary, in warm water at 40 °C to 70 °C.

Take the combined sodium hydroxide washings, including any acidified for the purpose of checking the washing procedure, which should be alkaline. Then boil the combined washings for 20 minutes, roughly maintaining the initial volume by the addition of water; a fragment (about 2 mm^3) of suitable inert material may be used to prevent bumping. Cool the sodium hydroxide washings to room temperature and, if clear, transfer them directly to the phenols flask (F.2.2) (see Note 5). If the solution contains suspended matter, filter it through glass wool (F.2.3) previously moistened with the sodium chloride solution (F.1.2) and collect the filtrate in the 200-ml phenols flask (F.2.2) (see Note 5); wash the glass wool with 25 ml of the sodium chloride solution (F.1.2) and add the latter to the filtered sodium hydroxide washings.

Add a few drops of the methyl orange indicator solution (F.1.3), slowly add the concentrated hydrochloric acid (F.1.4) until the methyl orange just indicates distinct acidity after mixing the two layers by swirling. During the addition of the hydrochloric acid, keep the contents of the flask cool by immersing the flask from time to time in cold water.

Add just sufficient powdered sodium chloride (F.1.5) to saturate the aqueous layer and leave a few particles undissolved. Shake to ensure thorough mixing and then stand to allow separation. During this process the phenols will separate as an upper liquid layer. Bring the phenols into the graduated portion of the flask by adding the saturated sodium chloride solution (F.1.2). After adequate setting time, preferably overnight, measure the volume of phenols (see Note 6).

NOTES

1. *A known mass (approximately 100 g) of dehyrated creosote will have been distilled to produce the combined fractions ($f_1+f_2+f_3+f_4$) which constitute this test portion.*

2. *Completion of the removal of phenols can be verified by slightly acidifying the last washings using the concentrated hydrochloric acid (F.1.4) and examining for separated phenols.*

3. *As a rough guide, it may be assumed that 25 ml of the sodium hydroxide solution of the strength indicated (F.1.1) are sufficient to remove about 5 ml of phenols.*

4. If necessary the upper layer may be diluted with toluene to secure a satisfactory separation.

5. If it is expected that the sample for test contains only small amounts of phenols, it is preferable to use about half the specified volumes of sodium hydroxide solution for the successive washings and to collect the sodium hydroxide washings (after filtration through the glass wool, if necessary) in the 150-ml phenols flask.

6. For some creosotes, the measurement of the liberated phenols is difficult because of their viscous nature. This may be overcome by adding a measured volume of toluene to the phenols flask immediately before the final addition of the saturated sodium chloride solution. From the observed volume of the separated upper layer in the phenols flask, subtract the volume of toluene added.

F.4 CALCULATION

$$\text{Extractable phenols, in the distillate upto 315 °C, ml/100g of dehydrated creosote} = \frac{V_o \times 100}{m}$$

where,

V_o is the volume, in millilitres, of phenols obtained in the test; and

m is the mass, in g, of the dehydrated creosote used for the test (in E.2.3).

APPENDIX G
DETERMINATION OF EXTRACTABLE PHENOLS BOILING UP TO 220 °C

NOTE

This examination of the extractable phenols is only applicable if the results obtained by the test described in Appendix F exceed 4 ml/100 g of dehydrated creosote.

G.1 REAGENTS

G.1.1 **Sodium hydroxide,** 100 g/l solution.

G.1.2 **Toluene.**

G.1.3 **Sodium chloride,** saturated solution.

G.1.4 **Methyl orange indicator,** 1 g/l solution.

G.1.5 **Hydrochloric acid,** concentrated (rel.den. = 1.18).

G.1.6 **Sodium chloride,** powdered solid.

G.2 APPARATUS

G.2.1 **Separating funnels**, stoppered, of appropriate capacity.

G.2.2 **Distillation apparatus**, described in **E.2.1** except that a 130-ml distillation flask is used in place of the 150-ml flask (item a of E.2.1).

G.2.3 **Measuring cylinder**, 100-ml capacity .

G.2.4 **Glass wool.**

G.3 PROCEDURE

Calculate from the result obtained in Appendix F, the mass of dehydrated creosote (see Appendices E and F) necessary to provide 120 ml of extracted phenols.

Transfer this quantity of dehydrated creosote to a separating funnel of appropriate capacity (G.2.1).

Rinse the vessel in which the creosote sample was measured with 300 ml of the sodium hydroxide solution (G.1.1), and add it to the contents of the separating funnel. Shake the funnel vigorously for 5 minutes and allow it to stand until separation is complete. If seperation proves difficult, reject the sample. Take a second sample and dilute it with an equal volume of toluene (G.1.2) before recommencing the procedure. Run the lower alkaline layer into a beaker.

Treat the upper layer of oil a further three times as above, using 100 ml of the sodium hydroxide solution each time. Combine all the sodium hydroxide washings and boil them vigorously for 10 minutes maintaining roughly the initial volume by the addition of water. Bumping may be minimized by the addition of fragments of inert porous material.

Cool the washings to room temperature and, if they are not clear, filter them through glass wool previously moistened with the saturated sodium chloride solution (G.1.3). Add a few drops of the methyl orange indicator (G.1.4) and then slowly add sufficient concentrated hydrochloric acid (G.1.5) to produce a distinct acidity after vigorous agitation. Add sufficient powdered sodium chloride (G.1.6) with shaking to saturate the aqueous layer and leave a few particles undissolved.

Transfer the whole mixture to the separating funnel, rinse the beaker with a small quantity of the saturated sodium chloride solution (G.1.3) and add this to the funnel to ensure complete transfer of the phenols. After allowing sufficient time to settle, run off the lower or aqueous layer and then transfer the remaining phenols to the 250-ml distillation flask (E.1.1.1.a).

Distil the contents of the flask slowly, collecting the distillate in a small separating funnel until all the water has distilled (as shown by the distillate becoming clear at the end of the condenser). Stop the distillation.

Add sufficient powdered sodium chloride (G.1.6) to the separating funnel to saturate the aqueous layer and leave a few particles undissolved after shaking. Allow to settle, run off the lower aqueous layer and return the phenols to the distillation flask. Mix well and allow to cool to room temperature. Measure 100 ml of the dry crude phenols into the 130 ml distillation flask. Attach the flask to the condenser and insert the thermometer.

Distil the contents of the flask at a rate of about one drop per second until a corrected temperature equivalent to 220 °C is reached. Apply the correction as described in E.2.1.2. Change the receiver, then stop the distillation. Read the volume of distillate obtained .

G.4 CALCULATION

$$\text{Extractable phenols boiling upto 220 °C, ml/100 g of dehydrated creosote} = \frac{\frac{V_2}{V_1} \times 100 \quad V_0}{m} \times 100$$

where,

V_0 is the volume, in ml, of extractable phenols measured in F.3.2;
V_1 is the volume, in ml, of dry crude phenols used for the test in G.3;
V_2 is the volume, in ml, of phenols boiling below 220 °C as determined in G.3; and
m is the mass, in g, of dehydrated creosote taken in E.2.3.

APPENDIX H
DETERMINATION OF KINEMATIC VISCOSITY

H.1 APPARATUS

H.1.1 **Test sieve,** nominal aperture size 75 um, complying with CS 124.

H.1.2 **Thermometer** , total immersion type.

H.1.3 **Water bath,** thermostatically controlled, capable of adjustment to 40 ± 0.1 °C. Ensure the temperature control is such that the temperature of the bath during the period of measurement does not vary from the desired temperature by more than 0.01 °C over the length of the viscometer or between viscometers. Ensure the depth of the bath is such that, when the viscometer complete with sample is in position, no part of the sample is less than 20 mm below the surface of the water or less than 20 mm above the bottom of the bath.

H.1.4 **U-tube reverse flow viscometers,** as follows with certificate of calibration :

a) viscosity range 2 mm^2/s to 10 mm^2/s and nominal factor of 0.01 mm^2/s^2; and

b) viscosity range 6 mm^2/s to 30 mm^2/s and nominal factor of 0.03 mm^2/s^2.

H.1.5 **Viscometer holder,** to hold the viscometer firmly in the thermostatic bath in the alignment stated in the certificate of calibration.

H.1.6 **Timing device,** graduated in divisions of 0.2 s or less and having an accuracy of 0.07 per cent over 15 minutes.

H.2 PROCEDURE

H.2.1 Preparation of test portion

Warm the sample (see Appendix B) to 45 °C and filter a minimum of 20 ml through the test sieve (H.1.1).

H.2.2 **Filling the viscometer** (see Figure 3).

Mount the thermometer (H.1.2) in the bath (H.1.3) vertically so that the top of the mercury column is within 2 mm of the surface of the water. Mount the dry clean viscometer (H.1.4) in the viscometer holder (H.1.5) in the bath in the alignment stated in the certificate of calibration and allow the viscometer to reach the bath temperature. Close Tube L (see Figure 2) with a rubber bung fitted with a stopcock or similar device so that the air can be prevented from escaping. With the stopcock open pour sufficient of the prepared sample (H.2.1) into the filling Tube N to a point just below the upper filling Mark H, avoiding wetting the glass above H. Allow the sample to flow through the capillary Tube R, taking care that the sample column remains unbroken until it has reached a position about 5 mm below the lower filling Mark G, arrest its flow at this point by closing the Tube L.

Add more of the sample to the filling Tube N to bring the upper surface of the sample to just below the Mark H. Keep the viscometer in the bath for at least 30 minutes to allow the sample to reach the bath temperature and any air bubbles to rise to the surface. Using the stopcock, carefully adjust the lower level of the sample so that its ring of contact with the glass is coincident with the bottom of Mark G. Add further sample to Tube N until the ring of contact of the creosote in the tube is coincident with the bottom of Mark H. Allow a little time for this additional amount of sample to reach the bath temperature.

H.2.3 **Measurement**

Remove the rubber bung from Tube L or open the stopcock to allow the sample to flow under its own head. Measure the time in seconds for the uppermost ring of contact of the sample with the glass to rise from the bottom of the lower Mark E to the bottom of the upper Mark F. Record the thermometer reading at the beginning, during and at the end of the timed flow.

NOTE

As some of the sample may remain on the walls of the viscometer between the timing marks, repeat determinations of flow time may be made only after emptying, cleaning and drying the viscometer, refilling it and then repeating the flow time measurement.

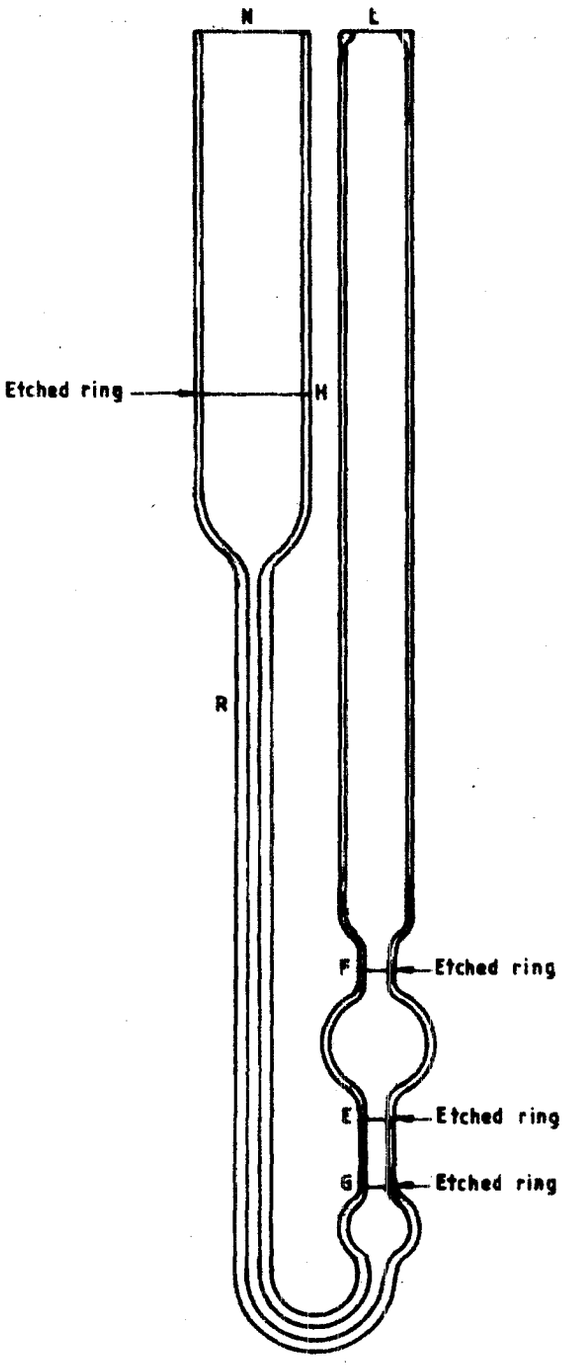

FIGURE 3 - U-tube reverse flow viscometer

H.3 CALCULATION

Kinematic viscosity, mm^2/s = Ct

where

C is the viscometer constant; and
t is the mean time of slow, in seconds.

APPENDIX J
DETERMINATION OF WATER CONTENT

J.1 REAGENT

Toluene or xylene, water saturated.

J.2 APPARATUS

J.2.1 **Measuring cylinder**, 100-ml capacity.

J.2.2 **Round-bottom flask**, about 500-ml capacity.

J.2.3 **Dean and Stark condensing and collecting system**, employing a 2-ml receiver.

J.3 PROCEDURE

Fill the measuring cylinder (J.2.1) to the 100-ml mark with the thoroughly mixed sample (see Appendix B) at room temperature, or at the lowest temperature of complete liquidity if that is above room temperature. Transfer the test portion to the flask (J.2.2). Wash the measuring cylinder with successive quantities of either toluene or xylene, 100 ml in all and add the washings to the flask. Add a fragment of porous inert material and connect the flask to the Dean and Stark system (J.2.3).

Heat the flask so that the condensate falls from the end of the condenser at a rate of 2 to 5 drops per second. Continue the distillation until the volume of water collected remains constant. Note the volume in millilitres of water in the graduated tube.

If a persistent ring of condensed water in the condenser tube is noted, clean the condenser and repeat the determination.

J.4 CALCULATION

Water content, per cent by volume = V

where,

V is the volume, in ml, of water in the graduated tube.

APPENDIX K
DETERMINATION OF MATTER INSOLUBLE IN TOLUENE

K.1 REAGENT

Toluene.

K.2 APPARATUS

K.2.1 **Filter crucibles,** glass or porcelain, 30-ml capacity, porosity grade P16.

K.2.2 **Oven,** capable of maintaing at 105 ± 5 °C.

K.2.3 **Desiccator,** with silica gel desiccant.

K.2.4 **Stainless steel filter,** mesh size 150 um, complying with CS 124.

K.2.5 **Beakers,** 250-ml capacity, with glass covers.

K.2.6 **Boiling water bath.**

K.3 PROCEDURE

Dry the crucible (K.2.1) in the oven (K.2.2) at 105 °C, cool it in the desiccator (K.2.3) and weigh it to the nearest 0.1 mg. Repeat the drying, cooling and weighing procedures until the difference between two successive weighings does not vary by more than 0.2 mg. Record the mass of the crucible.

Pour 25 g to 30 g of the well-mixed sample (see Appendix B) through the stainless steel filter (K.2.4) into the beaker (K.2.5) weigh to the nearest 0.1 g and record the mass of the sample. Add 100 ml of toluene to the beaker, cover the beaker with a glass cover and heat on the boiling water bath. Cautiously stir the contents of the beaker with a glass rod. When the sample has dissolved, cover the beaker and leave on the water bath for about 10 minutes to allow the greater part of the insoluble matter to settle.

Heat approximately 200 ml of tolune on the water bath to 80 °C to 100 °C to be used for washing purposes.

Decant the supernatent solution in the beaker through the crucible using gentle suction to assist filtration. Using the hot toluene, quantitatively transfer the insoluble matter to the filter crucible and rinse out the beaker. Wash the filter and its contents three or four times using the whole of the remaining hot toluene.

Dry the crucible in the oven (K.2.2) at 105 °C, cool it in the desiccator (K.2.3) and weigh it to the nearest 0.1 mg. Repeat the drying, cooling and weighing procedures until the difference between two succesive weighings does not vary by more than 0.2 mg. Record this mass.

K.4 CALCULATION

$$\text{Matter insoluble in toluene, per cent by mass} = \frac{(m_3 - m_1)}{m_2} \times 100$$

where,

m_1 is the mass, in g, of the crucible;
m_2 is the mass, in g, of the test portion ; and
m_3 is the mass, in g, of the crucible and residue after drying.

APPENDIX L
DETERMINATION OF SATURATED HYDROCARBONS CONTENT

L.1 REAGENT AND MATERIAL

L.1.1 **Silica gel,** in the particle size range of 104 um to 500 um (see Note 3 to L.3), heated for 4 hours at 150 °C and stored in a sealed container until required.

L.1.2 **Light petroleum,** boiling range 40 °C to 60 °C, aromatic free.

L.2 APPARATUS

L.2.1 **Absorption column,** a glass tube about 650-mm long and of 10-mm to 10.5-mm internal diameter.

L.2.2 **Specimen tubes**, 75 mm x 25 mm, with a mark at the 5-ml level, serially numbered and tared.

L.2.3 **Boiling water bath.**

L.2.4 **One-mark volumetric flask,** 100-ml capacity .

L.3 PROCEDURE

Plug the constricted end of the absorption column (L.2.1) with a small plug of cotton wool. Pour 40 g of the silica gel (L.1.1) into the column in five or six portions; tap the column between the addition of successive portions in order to pack the gel. Continue the tapping as long as the level of the silica continues to fall in the column, and then insert a second cotton wool plug and press firmly down on to the surface of the gel.

Weigh, to the nearest 1 mg, about 2 g of the sample (see Appendix B) in a 50-ml beaker and add about 2 ml of the light petroleum (L.1.2). Pour this mixture carefully on to the column endeavouring to avoid splashing on the sides or creeping of the solvent. Rinse the beaker twice with 3-ml portions of the light petroleum and transfer the rinsings to the column when the original mixture has been absorbed. When the rinsings in turn have been absorbed, elute the column with 100 ml of the light petroleum.

Collect the eluate in 5-ml portions in the series of numbered and tared specimen tubes (L.2.2). During the filling of each tube, ensure that the delivery tube projects well down inside the specimen tube in order to minimize loss by evaporation. This is conveniently arranged by standing the specimen tube, during filling, on a support block which is momentarily withdrawn at each change of tube. When all the specimen tubes are filled to the 5-ml mark, leave them to stand on the boiling water bath (L.2.2) and assist evaporation of the solvent by blowing into them an occasional gentle stream of air from a rubber blow-ball.

Just before the last of the solvent disappears from each individual tube (see Note 1) remove the tube from the water bath and complete the evaporation by means of a gentle stream of air at room temperature. Wipe each tube on the outside and cool it in the desiccator before weighing (see Note 2).

Return to the water bath for 30 seconds, all tubes from the first in the sequence, to the first tube which contains less than 5 mg of residue. Direct a slow stream of air into each tube during the whole of that time. Cool and reweigh each tube as before and, if the new mass of any tube differs by more than 5 mg from the previous mass, return the tube to the bath for a further 30 seconds. Repeat the weighing, reheating and evaporating procedures until the difference between two successive weighings does not vary by more than 4 mg. Use the first of the last two consecutive weighings for the calculation. Total the masses of the residues in all the tubes up to and including the one which was found to contain less than 5 mg of residue (see Note 3).

NOTES

1. *The residues in all the tubes except the first one or two are normally very small and the tubes should be removed from the water bath when they appear very nearly empty. A further guide to the time at which the tube should be removed from the water bath is provided by the ring of condensed solvent which can be seen at first below the mouth of the tube. This ring disappears during each period of gentle blowing, but reappears when the air stream is stopped, as long as any quantity of solvent remains. When it fails to reappear after blowing, the tube should be removed.*

2. *It will be found that the masses of residues in the tubes decline steadily from the first in the sequence and reach a minimum.*

3. *If the silica gel is of satisfactory activity, the mass of at least one of the residues should be less than 1 mg. If it is not so, the determination should be repeated using a different batch of gel.*

L.4 CALCULATION

$$\text{Saturated hydrocarbons content, percent by mass} = \frac{m_2}{m_1} \times 100$$

where,

m_1 is the mass, in g, of the test portion ; and
m_2 is the mass, in g, of the total residues obtained in L.3.

APPENDIX M
DETERMINATION OF NAPHTHALENE CONTENT

M.1 REAGENTS AND MATERIALS

Check all reagents for purity by passing a sample through the chromatograph under the conditions of the determination (see M.5). If a response is obtained on the chromatogram that is likely to cause significant errors in the determination on the test sample (specific details are given later in the text), reject the reagent or material concerned.

M.1.1 **Naphthalene,** with a minimum melting point of 79.6 °C.

M.1.2 **1,2,3,4-tetrahydronaphthalene,** (tetralin), of such purity when analysed by chromatography it shows no peaks that would coincide with those of naphthalene.

M.1.3 **Toluene,** 1 ul, (see Note) when analysed by chromatography, shall show no peaks that would coincide with those of napthalene or tetralin.

NOTE

Some sample splitters may require a different injection volume and this should be balanced with the procedure given in M.3.3.1.3.

M.1.4 **2,3-benzothiophene, (thionaphthalene),** of such purity, when analysed by chromatography it shows no peak between those of napthalene and 2,3 benzothiophene.

M.2 APPARATUS

M.2.1 **Volumetric flask,** one mark, 10-ml capacity.

M.2.2 **Gas chromatograph,** fitted with a flame ionization detector and heated injection port with sample splitter and having the following characteristics :

a) column temperature range of 130 °C to 200 °C ;
b) injection port temperature set at 200 °C to 250 °C;
c) silica capillary column,approximately 25 m in length, 0.2mm internal diameter, coated with methyl silicone gum ; and
d) potentiometric strip chart recorder with minimum chart width of 200 mm and maximum response time of 1 second or a data processor.

NOTE

An electronic integrator may be used as an addition or as an alternative in M.3.3.2. to M.3.3.4.

M.2.3 **Micro-pipette, syringe type,** suitable for accurately injecting 1 ul portions into the gas chromatograph.

M.2.4 **Micro-pipette, syringe type,** capable of accurately measuring 100 ul.

M.2.5 **Analytical balance.**

M.3 PROCEDURE

M.3.1 Preparation of calibration solutions

Prepare duplicate calibration solutions of approximately the same composition by the following procedure :

Weigh, to the nearest 0.000 2 g, approximately 0.1 g of the naphthalene (**M.1.1**) into the volumetric flask (**M.2.1**) (including the flask stopper in the weighing). Add carefully from a syringe 10 ul of the 1,2,3,4-tetrahydronaphthalene (**M.1.2**), stopper the flask and reweigh. Dilute to the mark with toluene (**M.1.3**).

M.3.2 Preparation of column performance solution

Weigh, approximately 0.01 g of the naphthalene (**M.1.1**) and 0.10 g of the 2,3-benzothiophene (**M.1.4**) into the volumetric flask (**M.2.1**). Dilute to the mark with toluene.

M.3.3 Determination

Throughout this procedure all solutions are analysed by chromatography by injecting 1 ul into the gas chromatograph, but see note to **M.1.3**.

M.3.3.1 *Setting up the gas chromatograph*

M.3.3.1.1 Oven conditions

For all analyses maintain the column oven at 130 °C until the 2,3-benzothiophene has been eluted. Then raise the oven temperature to 200 °C (either manually or by a temperature programmer) and maintain at this temperature until the rest of the sample has been eluted. Then reset the oven to 130 °C and allow sufficient time for the oven to reach equilibrium at this temperature before running the next sample.

M.3.3.1.2 Carrier gas

Adjust the carrier gas flow to give a retention time for naphthalene of 15 minutes to 20 minutes.

M.3.3.1.3 Detector signal

Adjust the detector signal to give a napthalene peak height of 50 per cent to 99 per cent of full scale deflection if using a strip chart recorder when analysing the calibration solution by chromatography (**M.3.1**).

M.3.3.1.4 Column performance

Satisfy the following criteria before the column is used for analysis. These criteria are checked following the necessary adjustment to the chromatograph described in **M.3.3.1.1, M.3.3.1.2** and **M.3.3.1.3.**

a) that peaks to be measured are symmetrical, i.e. show no obvious tailing;

b) that the number of theoritical plates measured on the naphthalene peak is not less than 5 000.
The theoretical plate number (n) is calculated from the equation;
$n = 16 \text{ (retention time/peak width)}^2$
where both retention time and peak width are measured in length terms directly from the recorder chart;

c) that the separation between napthalene and 2,3-benzothiophene is such that the trough ratio is less than 0.2. The trough ratio is determined by dividing the height of the trough between two partially resolved peaks by the height of the smaller of the two peaks both being measured from the baseline.

M.3.3.2 *Calibration*

Analyse in turn 1 ul of each of the duplicate calibration solutions (**M.3.1**) by chromatography using the conditions described in **M.3.3.1** to obtain separate chromatograms.

Measure the heights of naphthalene and 1,2,3,4-tetrahydronaphthalene peaks from the resultant chromatograms (or take the equivalent values from an integrator).

Calculate the calibration factor f_n from the formula :

$$f_n = \frac{H_t}{H_n} \times \frac{m_n}{m_t}$$

where,

H_t is the peak height of 1,2,3,4-tetrahydronaphthalene (or integrator reading);
H_n is the peak height of naphthalene (or integrator reading);
m_t is the mass, in g, of 1,2,3,4-tetrahydronapthalene in 10 ml of solution; and
m_n is the mass, in g, of naphthalene in 10 ml of solution.

Determine the mean of the two factors.

M.3.3.3 *Analysis*

If the test sample (see Appendix B) required warming to dissolve solid matter before analysis, care should be taken to avoid losing volatile constituents.

If the approximate naphthalene content is not known, weigh out 0.20 g of the test sample (see Appendix B), dilute to 10 ml with toluene (M.1.3) in the volumetric flask (M.2.1) and analyse 1 ul by chromatography using the same conditions as for the calibration (M.3.3.2). Measure the peak height for naphthalene and compare this with the peak height for the concentration values obtained from the calibration solutions. Calculate the approximate naphthalene content.

Weigh a test portion containing 0.05 g to 0.1 g of naphthalene into a tared volumetric flask (M.2.1) and dilute to the mark with toluene. Inject 1 ul of this solution into the chromatograph and obtain the second chromatogram.

M.3.3.4 *Measurement of peak heights*

On the chromatogram draw the peak base for each relevant peak. Record the peak heights for napthalene and 1,2,3,4-tetrahydronaphthalene in the second chromatograph, and for napthalene and any coincident peak with the 1,2,3,4-tetrahydronaphthalene retention time in the first chromatograph.

NOTE

The peak base is defined as the interpolation line drawn between the start and the finish of the peak and represents the base line that the chromatogram would have followed if the sample component responsible for the peak had not been present. Measure the perpendicular distance between the apex of the peak and the peak base.

M.4 CALCULATION

$$\text{Napthalene content, per cent by mass} = \frac{(H_{n1} + H_{n2})}{2\,m_s} \times \frac{m_t}{(H_{t2} - H_{t1})} \times 100\, f_n$$

where,

f_n is the calibration factor;
m_t is the mass, in g, of 1,2,3,4-tetrahydronaphthalene added;
m_s is the mass, in g, of sample taken;
H_{n1} is the height, in mm, of naphthalene peak (or integrator reading) for the second chromatogram;
H_{n2} is the height, in mm, of naphthalene peak (or integrator reading) for the second chromatogram;
H_{t1} is the height, in mm, of peak at 1,2,3,4-tetrahydronaphthalene retention time (or integrator reading) for the first chromatogram; and
H_{t2} is the height, in mm, of 1,2,3,4-tetrahydronaphthalene peak (or integrator reading) for the second chromatogram.

APPENDIX N
FIRST-AID TREATMENT

N.1 SKIN

The affected area may be washed immediately with industrial methylated spirit, followed by a wash with soap and water.

N.2 EYE

N.2.1 Immediate treatment is very important. Eye/eyes may be washed thoroughly with running cold water. Alternatively, if quick application is possible, use copious quantities of buffered phosphate solution prepared as in N.2.2.

N.2.2 Phosphate solution for first-aid treatment

Mix 700 g of anhydrous potassium di-hydrogen phosphate ($KH_2PO_4.12H_2O$) in 850 ml of distilled water. (The solution can be stored for 3 months only). For use, dilute it with three times of water.

N.3 Follow up with medical advice.

SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI *which includes surveillance inspection of the factory, testing of both factory and market samples.*

Further particulars of the terms and conditions of the permit may be obtained from the Sri *Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.*

Printed at SLSI (Printing Unit)

SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.

Printed at the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.