SRI LANKA STANDARD 1109 : PART 2 : 1995

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SPECIFICATION FOR TIMBER PRESERVATION BY MEANS OF COPPER/CHROME/ARSENIC COMPOSITIONS

PART 2 : TEST METHODS

SRI LANKA STANDARDS INSTITUTION

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SLS 1109: Part 2 : 1995

Gr. 12

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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 TIMBER PRESERVATION BY MEANS OF COPPER/CROME/ARSENIC COMPOSITIONS

PART 2 : TEST METHODS

FOREWORD

This standard was approved by the Sectoral Committee on Timber and Timber Based Products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1995-12-14.

With the industrial and agricultural development of the country coupled with increased construction activity, the demand for timber for various purposes has increased considerably. Due to the limited availability of naturally durable timber species, timber supply has to be augmented by selected timber species of lesser durability which, when suitably preservative treated, would give adequate service life Most imported timber also need treatment. Hence, preservative treatment of timber forms a very important part of the national effort to conserve the natural resources of the country, and to achieve their most economic utilization. Already many new treatment plants are being planned to increase, substantially, the treatment capacity locally.

Copper/chrome/arsenic (CCA) preservative is widely used for treatment of timber. It has also proved effective for treating a wide range of species for a variety of applications due tothe following favourable considerations: (a) The solvent water is readily available at negligible cost; (b) Required retentions can be easily attained by varying the concentration of the treating solution; (c) Evaporation is negligible; (d) The preservative odourless; (e) Economy in freight due to availability in powder or paste form; (f) Easy penetration into timber due to non-viscous and non-oily constitution; (g) Amenability of treated timber to painting, polishing and gluing; and (h) Possibility of overcoming its toxicity to animals and humans by adopting proper precautionary measures.

The efficacy of preservative treatment of timber depends on the quality of the preservative, and also the treatment process which ensures the attainment of the required absorption and penetration of the preservative into the timber. Hence a Sri Lanka standard on this subject was considered useful to safeguard the interests of both the consumer and the preserver as well as the overall safety of the operation.

This standard is subject to the provisions of the Control of Pesticides Act No 33 of 1980 and the regulations framed thereunder.

This part of the standard (Part 2) specifies the test methods. Part 1 of this standard specifies the compositions of the preservative, the methods of application,the retentions and penetrations desired from the prescribed treatment, and a method for assessing the effectiveness of treatment.

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

The Sri Lanka Standards Institution gratefully acknowledges the use of the publications of the British Standards Institution, the Bureau of Indian Standards, the Standards and Industrial Research Institute of Malaysia, and the South African Bureau of Standards.

1 SCOPE

This part of the standard specifies test methods related to the preservative treatment of timber by means of water-borne copper/chrome/arsenic compositions.

2 REFERENCES

ISO	3696	-	Water for laboratory use
BS	700	-	Graduated pipettes
BS	846	-	Burettes
BS	1792	-	Volumetric flasks
BS	2648		Performance requirements for electrically heated laboratory drying ovens
SLS	127		Test sieves
SLS	985	-	Grading of Timber Part 1 - Species of timber
SLS	1109		Timber preservation by means of copper/chrome/arsenic compositions Part 1 - Treatment processes

3 DEFINITIONS

3.1 **penetration** : The depth to which preservative enters the timber.

3.2 **pH value** : The negative decimal logarithm of hydrogen-ion concentration in moles per litre, giving measure of acidity or alkalinity of a solution.

3.3 **spectro-photometer** : An instrument for measuring intensity of light in various parts of the spectrum.

3.4 **thief tube** : A device for the purpose of sampling the preservative in a drum.

3.5 **triangulation method of sampling** - Taking samples from the vertices of a triangle drawn at random on the surface of the material.

4 METHODS OF SAMPLING

4.1 Selection of drums from a consignment

4.1.1. The least number of drums to be sampled out of any given consignment of preservative material, irrespective of whether it is composed of a solid or a paste, shall be the nearest whole number to the square root of half the total number of drums in the consignment (see **Table 1**). The drums to be sampled shall be taken at random.

TABLE 1 - Sampling of drums

Number of drums in consignment (1)	No. of drums assumed(x) (2)	$\left\{\frac{x}{2}\right\}^{1/2}$ (3)	Number of drums to be sampled (4)
up to 10	10	2.24	2
11 to 20	20	3.16	3
21 to 50	50	5.0	5
51 to 100	100	7.07	7

4.2 Sampling the preservative

4.2.1 *Preservatives in dry powder form*

4.2.1.1 Apparatus

A standard "thief tube" of internal diameter 32 mm and probe length 900 mm is a suitable sampling device for the purpose of sampling the quality of the preservative in a drum.

4.2.1.2 Procedure

Take three "thief" samples by the triangulation method from each drum selected for testing. Transfer the contents of the "thief" to an airtight sample container of about 2.25 kg capacity. Bulk the extracted samples and mix well. Transfer the entire sample to a clean dry surface and heap into a cone. Turn over to form a new cone carrying out this operation three times. Form each new conical heap by depositing material on the apex of the cone so that the portions which slide down the side are distributed, as evenly as possible, and that the centre of the cone is not displaced. Some of the larger aggregates of the mixture may roll and scatter round the base, and these should be pushed back to the edge of the heap or broken and distributed evenly over the heap. Flatten the third cone from the mixed contents of the container by repeated vertical insertions of the edge of a board, commencing about the centre and working radially round the cone, lifting the board clear of the material after each insertion. Carry out this operation so that the flattened heap is of uniform thickness and diameter, and the centre coincides with the centre of the original cone.

Quarter the heap along two diameters which interesect at right angles, using a suitable divider. Shovel one pair of opposite quarters into a heap and reject the remainder. Mix and cone three times as described above, flatten the cone and quarter along two diameters. Repeat these operations until about 200 g of gross sample remains. Take utmost care to reduce to a minimum the moisture picked up during the sampling and mixing and reducing processes. If necessary, grind the whole of the gross sample to pass a No. 8 (2 mm) mesh test sieve and mix the ground sample well. Immediately enclose the test sample in an air-tight container until ready for analysis.

4.2.1.3 Preparation of solution (20 g/l) for analysis

Dissolve 40.0 g of the ground sample in distilled water, in a beaker. Filter into a 2000 ml volumetric flask, wash the residue carefully and make up to volume.

4.2.2 *Preservatives in paste form*

4.2.2.1 Apparatus

a) Small weighted sampling can of 500 ml capacity

It should be of such weight as to sink readily in the material to be sampled. The can should be fitted with a handle to which is attached a chain and with a removable stopper or cap to which is attached a separate chain so that after lowering the can to the required depth in the paste, the stopper can be removed and the container allowed to fill.

B) A stout steel stirring rod, suitable for stirring the contents of the drum.

4.2.2.2 Procedure

Thorough mixing of the preservative in each drum is necessary before samples are taken. Any settled material should be displaced from the base of the drums using the stirring rod. The drum closures should then be re-attached and the drums shaken and rolled to effect complete homogenisation of the contents. Mechanical agitation should be used if available. The drums should then reopened and examined for uniformity by probing with the steel rod. Alternate stirring, shaking and rolling should be continued until the contents are completely homogeneous.

Three samples are then taken from each drum with the sampling can, from just below the surface of the preservative, at a position about half-way between the surface and the base of the drum and from near the base. The three samples from each drum should be poured into a clean glass or plastic container and mixed together.

NOTE
Unless mixing is done as stated above, results may not be representative of the material used.

4.2.2.3 Preparation of solution (20 g/l) for analysis

Dissolve 40.0 g of the mixed sample in distilled water in a beaker. Filter into a 2000 ml volumetric flask, wash the residue carefully and make up to volume.

4.3 Extraction of the preservative from treated Timber

Ensure that the sample of treated timber taken is as representative as possible of the timber or timbers concerned. The area of the timber selected shall be free from end penetration; if this precaution is not taken, the results obtained will be too high.

Avoid samples consisting entirely of heartwood unless the timber under test is all heartwood, otherwise the results obtained will be too low. Similarly, avoid samples consisting entairly of sapwood unless the timber under test is all sapwood, otherwise the results obtained will be too high.

Pulverize about 20 g of the dry sample unless all the sample passes through 425 micron sieve. Collect the wood flour, mix well and dry to constant weight in an oven at 110°C.

Weigh approximately 5.00 to 10.00 g of the prepared wood flour and transfer to a Kjeldahl flask. Add concentrated nitric acid to the sawdust at the rate of about 8 ml per gram of the sawdust, and 5 ml of concentrated sulphuric acid for the entire mass and allow the contents to stand overnight under a hood. Gently heat for one hour and increase the temperature slowly. When charring begins add successive portions of 5 ml of concentrated nitric acid, until the solution becomes clear green. Allow to cool. Make it to a known volume with distilled water in a graduated flask. This solution should be used for the determination of arsenic , copper and chromium salt.

5 DETERMINATION OF pH VALUE

5.1 Apparatus

5.1.1 Sintered glass filter, of pore size index P 40.

5.1.2 Volumetric flask, one-mark, 500 ml capacity.

5.1.3 ph meter, with glass electrodes.

5.2 Procedure

5.2.1 Weigh, to the nearest milligram, a mass of the preservative equivalent to 10 $\pm$ 0.1 g of the nominal composition. Transfer this test portion to a beaker and dissolve in 250 ml of hot water, not exceeding 40°C, stirring continuously. Cool the solution to room temperature and filter it through the pre-weighed sintered glass

filter (5.1.1) into the volumetric flask (5.1.2). Wash the beaker and the residue on the filter five times with 10 ml portions of hot water not exceeding 40°C, collecting the washing in the flask. Dilute to the mark with water. { Reserve the filter and residue for the determination of insoluble matter, if required (see 6)}.

5.2.2 Determine the pH of the test solution at 27 ± 1°C using a pH meter.

6 DETERMINATION OF INSOLUBLE MATTER CONTENT

6.1 Apparatus

6.1.1 Sintered glass filter, of pore size index P 40.

NOTE

The sintered glass filter should be clean enough.

6.1.2 Oven, capable of maintaining a temperature of 105 ± 5° C.

6.2 Procedure

6.2.1 Weigh, to the nearest milligram, a mass of the preservative sample equivalent to 10 ± 0.1 g of the nominal composition and dissolve it in water. Filter and wash as described in 5.2.1 or, if the pH has already been determined, use the residue on the filter.

6.2.2 Transfer the sintered filter containing the residue to the drying oven (6.1.2) previously adjusted to 105 ± 5°C.Dry initially for a period of 1 hour, cool in a desiccator to room temperature and re-weigh. Repeat the drying, cooling and weighing operations at suitable intervals until the difference in mass between two successive weighings is not greater than 0.5 mg.

6.3 Calculation

$$\text{Insoluble matter, per cent by mass} = \frac{m_2 - m_1}{m_3} \times 100$$

where,

m_1 is the mass, in g, of the sintered glass filter;
m_2 is the mass, in g, of the sintered glass filter and residue after drying; and
m_3 is the mass, in g, of the preservative taken for the determination.

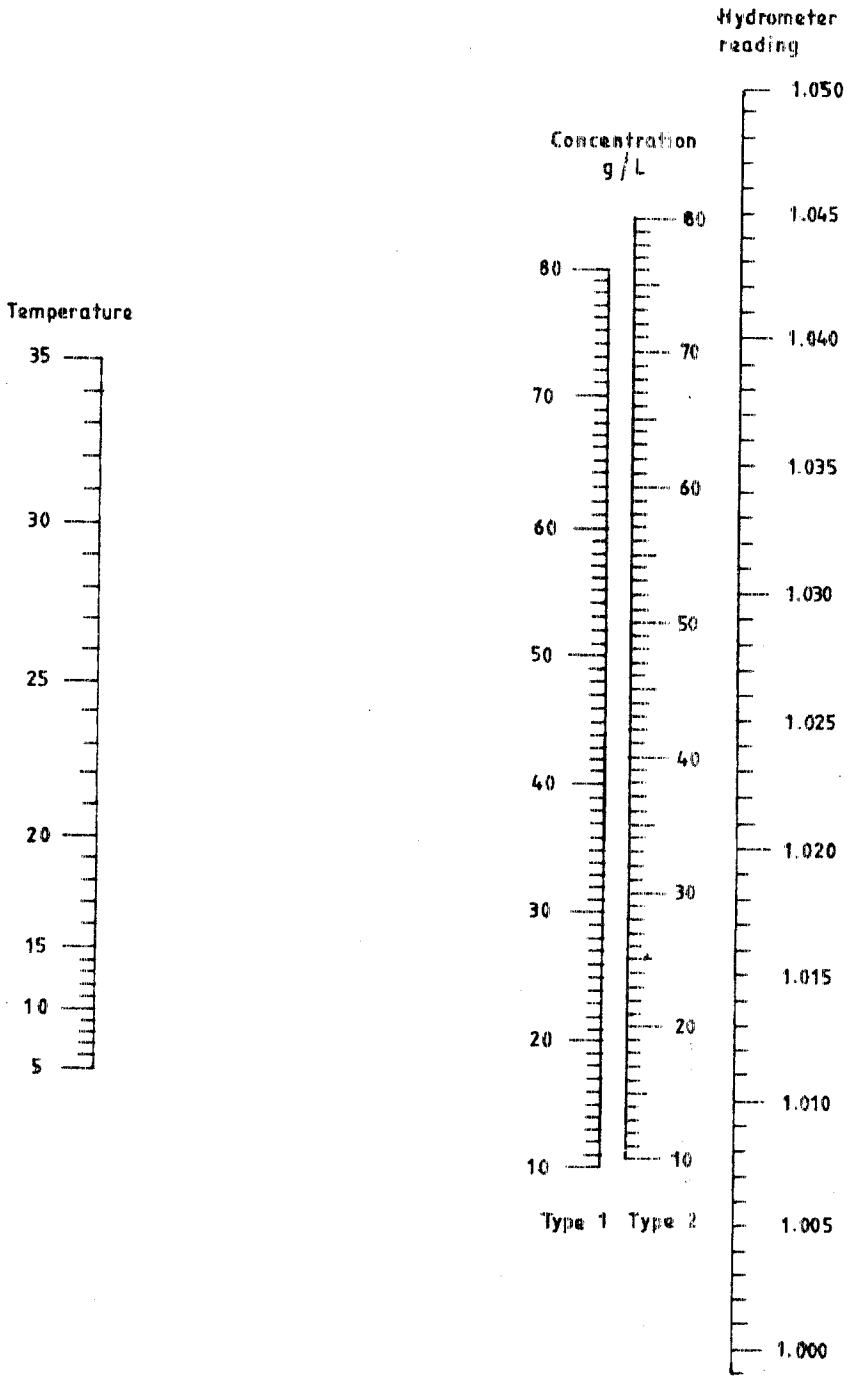

FIGURE 1 Hydrometer reading: concentration calibration chart for working solutions of type 1 and type 2 CCA preservatives

7 DETERMINATION OF THE CONCENTRATION OF THE WORKING SOLUTION

7.1 Apparatus

7.1.1 Density hydrometer, graduated to an accuracy of 0.0002 for medium surface tension.

7.1.2 Glass cylinder, 500 ml nominal capacity and overall height not greater than 300 mm.

7.2 Procedure

Determine the density of the solution at 27°C and read the corresponding concentration from Table 2, or Figure 1.

Obtain a representative 500 ml sample of the working solution.

Transfer the solution to the glass cylinder and place it on a level surface.

Insert the thermometer into the solution, wait until the temperature has reached equilibrium with its surroundings and remains constant for 1 min and record the temperature before removing the thermometer.

Place the clean, dry hydrometer in the solution and when the hydrometer has settled to a constant level, record the height of the solution level against the graduated scale on the stem of the hydrometer, with the eye directly in line with the solution to the nearest 0.005.

Using a hydrometer calibration chart the accuracy of which has been verified by analysis (see Figure 1), place a straight edged ruler across the temperature and hydrometer reading scales at the points recorded for the solution sample.

The concentration of the solution (in g/l) of the preservative type under test is read at the point of intersection on the concentration scale.

NOTE

The densities given in Table 2 apply to preservatives of the nominal compositions given in Table 1 of SLS 1109 : Part 1 : 1995. In practice, different tables will apply to concentrated or hydrated products.

Where the actual composition departs from the nominal composition, alternative density-concentration relationships are available from the manufacturers.

TABLE 2 - Density at 27°C as a function of concentration

Concentration of preservative	Density at 27°C	
	Type 1	Type 2
kg/m³ *	Mg/m³ **	Mg/m³ **
10	1.003	1.003
15	1.006	1.007
20	1.009	1.010
25	1.013	1.013
30	1.016	1.017
35	1.019	1.020
40	1.022	1.024
45	1.026	1.027
50	1.029	1.031
55	1.032	1.034
60	1.036	1.038
65	1.039	1.041
70	1.042	1.045
75	1.045	1.048
80	1.049	1.052

* 1 kg/m³ = 1 g/l
** 1 Mg/m³ = 1 g/ml

8 DETERMINATION OF COPPER CONTENT

An alternative method for determination of copper content is given in 11.

8.1 Reagents

The reagents shall be as listed below. They shall be of a recognized analytical reagent quality.

Distilled water complying with ISO 3696 shall be used.

a. Ammonium hydrogen difluoride
b. Ammonium thiocyanate
c. Potassium iodide, iodate-free
d. Methanol, redistilled using calcium oxide
e. Hydrochloric acid, concentrated, sp.gr.1.18
f. Sodium hydroxide, 100 g/l solution
g. Standard copper sulphate solution, containing 10 mg of $CuSO_4 5H_2O$ per ml.
h. Sodium thiosulphate solution
   Dissolve 9.94 g of sodium thiosulphate in a sufficient amount of water. Add 0.1 ml chloroform and dilute to 1000 ml. Store preferably in a dark bottle.

j. Starch solution

Mix 1.0 g of soluble starch with water to give a paste, pour the paste into boiling water, stirr and make up to 100 ml and boil for 1 min. Sterilize by adding 3 mg of mercuric iodide.

8.2 Procedure

Use the preservative solution, prepared as described in 4 for analysis.

Transfer 25 ml of the solution into a 500 ml conical flask. Add 10 ml of concentrated hydrochloric acid and then, with care, add 15 ml of methanol down the side of the flask.

Warm to boiling and continue heating until all the hexavalent chromium is reduced, as evidenced by the absence of any yellowish-green colour (see **Note 1**).

Wash down the sides of the flask with water and boil for 1 min. Cool and dilute to approximately 100 ml and add, from a burette, 10 per cent (100 g/l) sodium hydroxide solution with vigorous swirling until the first permanent precipitate forms.

Add concentrated hydrochloric acid dropwise until the solution becomes clear. Boil down to about 50 ml and cool to room temperature.

Add 3 g of ammonium hydrogen difluoride and shake well (see **Note 2**). The pH value of the solution before adding the potassium iodide should be 5.0 or slightly above if high results are to be avoided.

Add 2 g of potassium iodide, stopper and swirl until dissolved and store the solution for 5 min in the dark. The solution will then turn brown.

Titrate the liberated iodine with the sodium thiosulphate solution until the brownish colour of the iodine disappears. Then add 2 ml of starch solution, the colour will change to violet at this point. Continue the titration until violet becomes colourless.

Add 2 g of ammonium thiocyanate just before the end point to sharpen the colour change.

NOTES

1. The solution should not be evaporated to dryness, otherwise difficulty may be experienced in re-dissolving the trivalent chromium salts, and the determination may be vitiated.

2. The conical flasks used in this determination should be rinsed out without delay after the completion of the titration as they are liable to become etched through the use of ammonium hydrogen difluoride.

8.3 Calculation

Let the volume of the sodium thiosulphate solution used be V_1 ml.

Percentage of copoper sulphate ($CuSo_4 5H_2$) in the sample = $V_1 \times F \times 200$ % m/m

where,

F is the mass of copper sulphate $CuSo_4 5H_2O$ equivalent to 1 ml of sodium thiosulphate solution

The determination of F, the copper sulphate equivalent of the sodium thiosulphate solution, is carried out as follows :

Pipette 25 ml of the standard copper sulphate solution into a 500 ml conical flask, dilute to approximately 100 ml and follow the procedure given above, from the addition of 10 per cent sodium hydroxide solution.

$$\text{Then, } F = \frac{0.25}{V_2}$$

where,

V_2 is the mean volume (ml) of sodium thio-sulphate used in duplicate run; and

F is as defined above.

9 DETERMINATION OF HEXAVALENT CHROMIUM CONTENT

An alternative method for determination of chromium content is given in 11.

9.1 Reagent

The reagents shall be as listed below. They shall be of a recognized analytical reagent quality.

a. Orthophosphoric/ Sulphuric acid mixture
Add 280 ml of orthophosphoric acid (sp gr 1.75) into a 2000 ml volumetric flask and make up to volume with 50 per cent v/v sulphuric acid.

b. Ammonium ferrous sulphate solution
Dissolve 80 g of ammonium ferrous sulphate in water containing 50 ml sulphuric acid (50 per cent v/v) and dilute to 1000 ml with water.

c. Standard potassium dichromate solution
Dissolve 10.0 g of potassium dichromate in water and dilute to 1000 ml.

d. Barium diphenylaminesulphonate solution. Dissolve 0.20 g in 100 ml of water.

9.2 Procedure

Use the preservative solution, prepared as described in 4 for analysis.

Transfer 25 ml of the solution into a 500 ml titration flask, add 25 ml of orthophosphoric acid and sulphuric acid mixture and dilute to 200 ml with water.

To the above,pipette 25 ml of the ammonium ferrous sulphate solution and 1 ml of the barium diphenylaminesulphonate indicator solution. Titrate the mixture against the standard potassium dichromate solution until the solution becomes deep purple. Let the volume of potassium dichromate solution used be V_1 ml.

Pipette 25 ml of the ammonium ferrous sulphate solution into another 500 ml titration flask. Add 25 ml of mixed acids and 1 ml of the barium diphenylaminesulphonate solution. Titrate with the standard potassium dichromate solution. Let the volume of potassium dichromate solution used be V_2 ml.

9.3 Calculation

The percentage of hexavalent chromium in the sample $= F (V_2 - V_1)$ (m/m)

where,

F = 2.00 for $K_2Cr_2O_7$
2.026 for $Na_2Cr_2O_7 2H_2O$
1.781 for $Na_2Cr_2O_7$
1.358 for CrO_3

10 DETERMINATION OF ARSENIC CONTENT

An alternate method for determination for arsenic content is given in 11.

10.1 Reagents

The reagents shall be as listed below. They shall be of a recognized analytical reagent quality.

a. Hydrochloric acid, concentrated, sp gr 1.18
b. Sulphuric acid, concentrated, sp gr 1.84
c. Hypophosphorous acid, 300 g/l solution
d. Potassium bromate, 0.1505 N solution
   Dissolve 4.188 g of potassium bromate ($KBrO_3$) in 1000 ml of water (1 ml = 0.01 g $As_2O_5H_2O$).
e. Methyl orange indicator
   Dissolve 0.01 g of methyl orange in 100 ml of water.

Distilled water complying with ISO 3696 shall be used.

10.2 Procedure

Use the preservative solution prepared as described in 4 for analysis.

Transfer 50 ml of the solution into a 300 ml beaker and add 50 ml of the concentrated hydrochloric acid and 30 ml of the hypophosphorous acid solution. Mix thoroughly.

Cover and heat slowly to boiling and continue boiling gently for 15 min.

Filter the precipitate using a Gooch crucible with an asbestos pad. When using a Gooch crucible, care should be taken during the packing of the asbestos pad. Careless packing will result in loss of arsenic. Wash with water to remove all traces of hypohosphorous acid.

Dislodge the asbestos pad holding the precipitate and wash carefully into the original beaker (See Note 1).

Add 10 ml to 15 ml of the concentrated sulphuric acid and dissolve the precipitate by heating gently, over a naked flame, while swirling the mixture, until fumes are produced. Allow to fume strongly for 2 min to 3 min, then leave to cool.

Slowly, with continual swirling, add 50 ml of water. Remove the asbestos pad, or both the asbestos pad and the Gooch crucible and wash well. Dilute the solution to about 100 ml.

Add 10 ml to 15 ml of the concentrated hydrochloric acid and two drops of the methyl orange indicator (See Note 2).

Titrate immediately with the potassium bromate solution, stirring continuously until the solution becomes colourless or possibly pale yellow.

NOTES

1. If the precipitate and pad are not completely removed from the crucible, transfer the crucible also to the beaker.

2. In order to produce a sharp end point and obtain an accurate titration, the acidity with respect to hydrochloric acid should be not less than 1.2 N and not more than 3.5 N.

10.3 Calculation.

Let the volume of potassium bromate solution required be V ml. The percentage (m/m) of the arsenic pentoxide in the sample expressed as ($As_2O_5 2H_2O$) = 0.01 X V X 100 % (m/m)

11 DETERMINATION OF COPPER, CHROMIUM AND ARSENIC CONTENTS USING ATOMIC-ABSORPTION SPECTROPHOTOMETRIC METHOD

11.1 Apparatus

An atomic absorption spectrophotometer capable of operating under the conditions specified in Table 3 is required.

NOTE

For the determination of arsenic, a hollow cathode lamp of high spectral output should be used.

11.2 Reagents

All reagents shall be as listed below. They shall be of a recognised analytical reagent quality. Water complying with ISO 3696 shall be used.

11.2.1 *Sulphuric acid, 2 M*

Cautiously add, with stirring and cooling, 224 ml of sulphuric acid (sp.gr 1.84) to 1600 ml of water, cool, dilute to 2000 ml with water and mix.

11.2.2 *Sodium sulphate, 30 g/l solution*

Dissolve 30 g of anhydrous sodium sulphate in water and dilute to 1000 ml with water.

11.2.3 *Sulphuric acid, 0.5 M /Sodium sulphate 3 g/l, solution*

Add 250 ml of 2 M sulphuric acid to 100 ml of sodium sulphate (30 g/l) and dilute to 1000 ml with water and mix.

11.2.4 *Hydrogen peroxide, 300 g/l solution*

Dissolve 300 g of hydrogen peroxide in water, and dilute to 1000 ml with water.Alternatively, hydrogen peroxide of a known strength can be diluted with water, suitable to obtain a solution strength of 300 g/l.

11.2.5 *Standard solution*

(1 ml = 2000 ug of $CuSo_4.5H_2O$, 3000 ug of $K_2Cr_2O_7$, and 3000 ug of $As_2O_5.2H_2O$)

Dissolve 0.2000 g of copper sulphate pentahydrate ($CuSo_4.5H_2O$), in water and transfer the solution to a 100 ml one-mark volumetric flask (**BS 1792**). Dissolve 0.3000 g of potassium dichromate ($K_2Cr_2O_7$), in water,add 10 ml of 2 M sulphuric acid and 2 ml of 30 per cent (100 volume) hydrogen peroxide solution, boil, cool and transfer to the volumetric flask containing the copper sulphate solution. Dissolve 0.2233 g of arsenic trioxide (As_2O_3) by boiling in a solution containing 15 ml of 2 M sulphuric acid, 2 ml of 30 per cent (100 volume) hydrogen peroxide solution and 10 ml of water, cool and transfer to the volumetric flask. Add 10 ml of 30 g/l sodium sulphate solution to the volumetric flask, dilute to the mark with water and mix.

11.3 **Instrument setting and operation**

The instrument settings and operating conditions for the determination of copper, chromium and arsenic shall be as described in Table 3.

11.3.1 *Flame ignition procedure for operating the argon (entrained air) /Hydrogen flame with a propane burner.*

Connect the argon cylinder, via a reducing valve, to the air inlet. Similarly connect the hydrogen supply to the fuel inlet. Ensure that the gas lines (especially the hydrogen line) are free from leaks and that the spray chamber is flushed with hydrogen. Adjust the hydrogen flow to 1500 $cm^3\ min^{-1}$ and ignite the flame. Turn on the argon and adjust the flow to 5000 $cm^3 min^{-1}$.

NOTE

Great difficulty will be experienced in igniting the mixture if the argon and hydrogen are introduced together.

TABLE 3 - *Instrument setting and operating conditions for the detection of copper, chromium and arsenic.*

Instrument control	Copper	Chromium	Arsenic
Wavelength (nm)	324.8	357.0 or 429.0	193.7 or 197.2
Slit width (mm)	0.08	0.06 to 0.10	0.30
Lamp current (mA)	4	8	7
Scale expansion	up to X 10	up to X 10	up to X 10
Burner acetylene (cm)	10	10	10
Burner height (cm)	1.0	0.5	1.4
Acetylene flow rate (cm^3min^{-1})at a pressure of 70 kPa	1 000	1 800	---
Air flow rate (cm^3min^{-1}) at a pressure of 210 kPa	5 000	5 000	5 000
Hydrogen flow rate (cm^3min^{-1})at a pressure of 70 kPa	---	---	18 000
Argon flow rate (cm^3min^{-1}) at a pressure of 210 kPa	---	---	5 000

NOTE

Small differences in setting may be necessary with instruments of different manufacture.

11.4 **Preparation of calibration solutions**

From a burette, complying with requirements specified in BS 846, or graduated pipette, complying with the requirements specified for Type2 in BS 700, transfer aliquot portions of 0.5 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml and 7 ml, of the standard solution (11.2.4) to 100 ml one mark volumetric flasks, complying with the requirements specified in BS 1792, dilute to the marks with the 0.5 M sulphuric acid/3 grams per litre sodium sulphate (11.2.3) solution and mix.

11.5 Procedure for analysis of preservative solution prepared as described in 4

11.5.1 Transfer by pipette 2 ml of the 20 g/l solution to a 200 ml one-mark volumetric flask. Add 50 ml of 2 M sulphuric acid and 20 ml of 30 g/l sodium sulphate solution, dilute to the mark with water and mix.

11.5.2 Aspirate, using the operating conditions given in Table 3 the 0.5 M sulphuric acid/ 3 grams per litre sodium sulphate solution to obtain the blank absorbance, and a suitable range of calibration solutions followed by the sample solution. Check the calibration solutions after the last sample has been run. Plot calibration curves of ug/ml of copper, chromium and arsenic, expressed as copper Sulphate, potassium dichromate and arsenic pentoxide, against absorbance. Determine the contents of copper, chromium and arsenic in the test solutions by comparing the absorbance readings with the calibration curves and express the results as the percentages of copper sulphate, potassium dichromate and arsenic pentoxide in the solutions.

11.5.3 *Calculation*

The percentages, mass by volume, of either copper (expressed as $CuSO_4.5H_2O$) or chromium (expressed as $K_2Cr_2O_7$), or arsenic (expressed as $As_2O_5.2H_2O$) are given by the formula :

$$\frac{C}{100}$$

where, C is the concentration of salt in the test solution in ug/ml.

11.6 Procedure for analysis of copper, chromium and arsenic in treated timber.

11.6.1 Ensure that the sample of treated timber taken is representative of the batch of timber concerned. Select a suitable area from each sample which is free from "end penetration"; if this precaution is not taken the results obtained will be too high. Avoid samples consisting entirely of heartwood unless the timber under test is all heartwood, otherwise the results obtained will be too low. Similarly avoid samples consisting entirely of sapwood unless the timber under test is all sapwood, otherwise the results obtained will be too high.

11.6.2 *Preparation of sample*

Pulverize about 20 g of dry sample, for example in a beater mill or hammer mill, until all the sample passes a 420 um test sieve, complying with SLS 127. Collect the wood flour, mix well and dry to constant mass in an over at 110°C.

11.6.3 *Digestion of wood flour*

Accurately weigh approximately 0.5 g of the dried wood flour and transfer to a micro-Kjeldahl flask. Add 4.5 ml of a cooled mixture of 2 parts by volume of 30 per cent (100 volume) hydrogen peroxide solution and 1 part by volume of concentrated sulphuric acid. Transfer the micro-Kjeldahl flask to a digestion stand and, without shaking, warm gently. A vigorous wet combustion reaction will commence.

Continue the reaction until a clear solution is obtained. Heat the solution until charring occurs. Add a further two or three drops of hydrogen peroxide solution and continue heating until the solution is decolourized. If necessary, add further increments of hydrogen peroxide. Continue heating the decolourized solution until white fumes are evolved. Cool wash the solution into a 50 ml one mark volumetric flask, add 10 ml of 2 M sulphuric acid solution and 5 ml of 3 per cent sodium sulphate solution, dilute to the mark with water and mix.

11.6.4 Aspirate, using the operating conditions given in Table 3, the 0.5 M sulphuric acid /3 grams per litre sodium sulphate solution to obtain the blank absorbance, and a suitable range of calibration solutions followed, by the sample solution. Check the calibration solutions after the last sample has been run. Plot calibration curves of ug/ml of copper, chromium and arsenic, expressed as copper sulphate, potassium dichromate and arsenic pentoxide, against the absorbance. To determine the copper, chromium and arsenic, expressed as copper sulphate, potassium dichromate and arsenic pentoxide, contents of the test solutions, compare the absorbance readings with the calibration curves.

11.6.5 *Calculation of the percentage of metals*

The percentages by mass of copper (expressed as $CuSO_4.5H_2O$) chromium (expressed as $K_2Cr_2O_7$), and arsenic (expressed as $As_2O_5.2H_2O$) in the dry wood is given by formula :

$$\frac{C}{200\ m}$$

where,

C is the concentration of each salt in the test solution in ug/ml; and

m is the mass of wood flour taken in grams.

NOTE

The primary purpose of the method described is to provide a means of checking whether or not a given batch of timber has been significantly undertreated. For this purpose, expression of the results on a mass/mass basis is usually adequate.

11.6.6 *Calculation of the dry salt retentions*

The result may be expressed as kilograms per cubic metre if the density of the actual sample of timber is known; it is not satisfactory to take an average figure for the species of timber concerned. Adjustments for the moisture content of the timber are also necessary if the density figure does not apply to the dry material.

The retention of the toxic ingredient of the oven-dry wood(expressed in kg/m^3) shall be calculated from the formula:

$$\frac{T \times D}{100}$$

where, T is the percentage by mass of toxic ingredient; and
D is the oven-dry density of the wood in kg/m^3.

11.6.7 *Calculation of the density of oven-dry wood*

Dry a block of wood of dimensions 75 mm x 50 mm x 25 mm to constant mass in an oven maintained at 105 ± 5°C. Weigh the dried block and calculate the density.

12 DETERMINATION OF MOISTURE CONTENT OF TIMBER

12.1 Oven drying method

12.1.1 *Sample selection*

Cut a sample consisting of a full cross section not less than 300 mm from one end and 13 mm to 19 mm thick.

Alternatively, if it is not possible to cut the timber, take borings totalling not less than 8 g, not less than 230 mm from one end. Using a test borer consisting of a hollow auger and extractor, bore from the sapwood face to the centre of the section. For timbers having a smallest dimension of 150 mm or over, take borings to a depth of 25 mm or the full depth of the sapwood if this exceeds 25 mm.

Put the sample into a stoppered weighing bottle or other airtight container immediately after extraction if it cannot be weighed at once.

12.1.2 *Apparatus*

Ordinary laboratory apparatus together with the following.

12.1.2.1 Electrically heated drying oven, complying with BS 2648.

12.1.3 *Procedure*

Weigh the sample as soon as possible after cutting or extraction and place it in the oven (12.1.2.1) which has been already adjusted to a temperature of 103 ± 2°C.

Remove the sample periodically, cool in a dessicator and reweigh it. Dry the sample to constant mass such that the loss in mass for a drying interval of 6 h does not exceed 0.1%.

12.1.4 *Calculation*

Calculate the moisture content (A) as a percentage of the dry mass according to the following equation:

$$A = \frac{100\ (m_1 - m_2)}{m_2}$$

where,

m_1 is the mass in grams of the sample when wet; and
m_2 is the mass in grams after drying.

12.2 **Moisture meter method**

12.2.1 *Sampling*

Take components to be measured from random positions in the charge.

NOTE
The number of heartwood and sapwood faces to be sampled should reflect the relative proportions of these types of wood in the charge as a whole.

12.2.2 *Apparatus*

12.2.2.1 Moisture meter, electrical resistance type, provided with insulated electrodes and calibrated for the species of wood to be measured. It shall be capable of making an individual measurement with an error of not more than 2% (m/m) for moisture contents between 7% and 28% (m/m).Its range shall be about 0 to 40% (m/m).

12.2.3 *Procedure*

If the total number of components in the charge is n, take moisture readings on at least $(n/2)^{1/2}$ separate components. Measure the moisture content in the middle of each face. Drive the electrodes into the wood to a depth appropriate to the type of wood as given in Table 4, so that the line between the tips of the electrodes is in the direction of or perpendicular to the grain, in accordance with the instructions for the type of meter used.

Make at least three measurements in each measuring area 10 mm to 15 mm apart to avoid any accidental error due to electrodes penetrating an inner invisible defect in the wood.

The moisture content is given by the mean of the averages of the three measurements on each face.

TABLE 4: Penetration depth of measuring electrodes

Type of timber	Depth (mm)
(a) Sapwood	25,or sapwood thickness if less than 25
(b) Heartwood of wood species other than those listed in (c) Post Other components	 25 12
(c) Heartwood of species like Hulanhik, Kirikon,Namandora, Galmandora, Siyambala and Halmilla (see SLS 985)	5

13 DETERMINATION OF PENETRATION

13.1 Principle

A solution of a reagent which produces a characteristic colouration in the presence of copper salts is brushed or sprayed on to a cross section of the treated timber to be tested. It has been shown that there is no significant difference between the penetration of the active ingredients of the preservative and the extent of the colouration produced. The latter can therefore be taken as a measure of the penetration of the preservative as a whole.

13.2 Selection of sample

Cut a cross section at least 450 mm from the end of the timber or, if this is not practicable, from the centre, and free enough from holes or notches to avoid the effect of end penetration. Cross sections cut from timber that has been re-dried after treatment may be tested immediately, but it is necessary to dry cross sections taken from freshly treated timber. Plane the surface of the cross section smooth. If it is not possible to plane the surface, free it from adhering sawdust.

If it is not permissible to cross-cut the timber,a rough indication of the penetration may be obtained from a boring made with an increment borer.

13.3 Method 1

13.3.1 Reagent

The reagents shall be of recognized analytical reagent quality.

1,5- diphenylcarbazide solution
Dissolve 0.5 g of 1, 5 - diphenylcarbazide in 15 ml of glacial acetic acid and add 125 ml of water.

Water complying with ISO 3696, shall be used.

NOTE

It is desirable that the solution should be freshly made just prior to testing.

13.3.2 *Procedure*

Spray the reagent solution (13.3.1) evenly over the surface to be tested. After a minute or two a reddish purple colour will appear where the preservative has penetrated. Examine the surface within 15 min from applying the reagent.

13.4 Method 2

13.4.1 Reagent

The reagents shall be of recognized analytical reagent qualitys.

Chrome Azurol Z reagent solution
Dissolove 0.5. g of chrome azuerol S and 5.0 g of sodium acetate in water and dilute to 100 ml.
Water complying with ISO 3696 shall be used.

13.4.2 Procedure

Spray the sample to be tested with the reagent mixture of chrome azurol S solution. The development of deep blue colour indicates the presence of copper. Untreted timber is coloured red.

Examine the surface within 15 min from applying the reagent.

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