SRI LANKA STANDARD 1012 : 1994

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SPECIFICATION FOR COPPER/CHROMIUM/ARSENIC BASED TIMBER PRESERVATIVES

SRI LANKA STANDARDS INSTITUTION

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SLS 1012 : 1994

Gr.11

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Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This standard does not purport to include all the necessary provisions of a contract.

SRI LANKA STANDARD

SPECIFICATION FOR COPPER/CHROMIUM/ARSENIC BASED TIMBER PRESERVATIVES

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.

The water-borne timber preservatives usually consist of a mixture of inorganic preservative salts together with a fixing agent, normally a dichromate, dissolved in water. This standard covers preservatives containing a mixture of compounds of copper(II), chromium (VI) and arsenic (V). The preferred mixtures of compounds are either copper(II) sulfate, sodium or potassium dichromate and hydrated diarsenic pentoxide or copper(II) oxide, chromium(VI) oxide and hydrated diarsenic pentoxide.

Water borne timber preservatives undergo chemical changes within the timber and become insoluble in water. Therefore, they are resistant to leaching and are suitable for both internal and external use. The treated timber is generally not corrosive to metals but should not be used in direct contact with light alloys or rubber. After re-drying, the timber is clean in appearance (although it may be coloured) and can be painted and glued satisfactorily. The preservative does not creep and stain adjacent materials.Treatment with these preservatives increases the moisture content of the timber and for some usages re-drying is necessary. The wetting of the timber during treatment and subsequent re-drying cause the timber to swell and then to shrink. This can result in raising of the grain and sometimes a certain amount of distortion when the quality of the timber is such as to render it prone to irregular movement. The normal range of water-borne preservatives can significantly increase the electrical conductivity of the timber but it does not give rise to any trouble once the timber has dried out. For users where low conductivity is important it is advisable to check that the treatment will not have any adverse effect.

This standard covers two mixtures of slightly different composition of copper/chromium/arsenic based timber preservatives. It may be supplied either in powder form or in paste or liquid form.

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. Health hazards associated with the preservative in powder form is greater when compared with the other forms. Therefore, from the safety point of view, it is recommended that use of powder form be avoided as far as possible. In case of an accident, first-aid treatment to be followed is given in Appendix **F** as a guidance.

For satisfactory performance of a timber preservative, not only the nature of the preservative but also its method of application to the timber is important.

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

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 SLS 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.

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.

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

BS 1282 : 1975	Guide to the choice, use and application of wood preservatives.
BS 4072	Wood preservation by means of copper/chromium/arsenic compositions
Part 1 : 1987	Specification for preservatives.
BS 5666	Wood preservatives and treated timber
Part 3 : 1991	Quantitative analysis of preservatives and treated timber containing copper/chromium/arsenic formulations.
MS 733 : 1981	Specification for copper/chrome/arsenic wood preservatives.

1 SCOPE

This specification prescribes the requirements and methods of test for water-borne timber preservatives consisting essentially of a mixture of compounds of copper(II), chromium (VI) and arsenic(V).

2 REFERENCES

SLS 102 Presentation of numerical values.
SLS 428 Random sampling methods.
SLS 692 Safety colours and safety signs.

3 TYPES

This specification covers two types of copper/chromium/arsenic based timber preservatives of slightly different composition, designated as Type 1 and Type 2.

4 REQUIREMENTS

4.1 Composition

4.1.1 The percentages of individual ingredients in the preservative shall be not less than the minimum values specified in Table 1, when tested by the methods prescribed in Column 7 of the table.

TABLE 1: Compositional requirements for copper/chromium/arsenic based timber preservatives

Sl. No. (1)	Ingredient (2)	Requirement for				Method of test (see note) (7)
		Type 1		Type 2		
		Nominal (3)	Minimum (4)	Nominal (5)	Minimum (6)	
i)	Copper, as $CuSO_4.5H_2O$, per cent by mass	32.6	29.5	35.0	31.5	Appendix B or Appendix C
ii)	Chromium, as $Na_2Cr_2O_7.2H_2O$, per cent by mass	41.0	37.0	45.0	40.5	Appendix B or Appendix C
iii)	Arsenic, as $As_2O_5.2H_2O$ per cent by mass	26.4	23.5	20.0	18.0	Appendix B or Appendix C

NOTE

An atomic absorption spectrometric method and a colorimetric method are prescribed for the determination of copper, chromium and arsenic in the preservative. One of these methods may be used.

4.1.2 The sum of the concentrations of the individual components shall be not less than 95 per cent (m/m) when determined by the methods prescribed in Appendix B or Appendix C.

4.1.3 In cases where water is added or removed, the ratio of the active ingredients shall be in the same proportion by mass as are the nominal compositions specified in Table 1, when tested by the methods prescribed in Column 7 of the table. The containers shall carry information on the mass of the preservative equivalent to unit mass of the nominal composition (see 5.2d).

4.2 pH value

The pH value of a solution equivalent to 20 g/l of the nominal preservative composition shall be not less than 1.8 and not more than 2.8 when determined as prescribed in Appendix D.

4.3 Insoluble matter

The insoluble matter content of the preservative shall be not more than 0.5 per cent by mass when determined by the method prescribed in Appendix E.

5 PACKAGING AND MARKING

5.1 The product shall be packed in clean, suitable containers. The containers shall be securely sealed to prevent the inadvertent escape of their contents.

5.2 The containers shall legibly and indelibly marked or labelled with the following:

a) Name of the product;
b) Type;
c) Safety sign to indicate toxic nature (see Figure 1) with colours and dimensions as given in SLS 692.
d) In cases where water is added or removed, mass of the preservative equivalent to unit mass of the nominal composition (see Table 1);
e) Net contents, in kilograms;
f) Name and address of the manufacturer and/or supplier (including the country of origin);
g) Batch identification mark.

FIGURE 1 - Safety sign to indicate toxic nature

6 METHODS OF TESTS

6.1 Tests shall be carried out as prescribed in Appendices B to E 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 schemes of sampling and inspection should be adopted.

A.1 LOT

All containers of copper/chromium/arsenic based timber preservatives of one type and belonging to one batch of manufacture or supply should constitute a lot.

A.2 SAMPLING APPARATUS

The following sampling apparatus or any other suitable instruments should be used when taking samples from a lot:

A.2.1 For preservatives in powder form

A standard "thief" of internal diameter 32-mm and probe length 900-mm.

A.2.2 For preservatives in paste or liquid form

A.2.2.1 *A weighted sampling can,* of such mass as to sink readily in the material to be sampled, and of about 600-ml capacity. It should be fitted with a long, stiff handle so that it can made to submerge in the material to be sampled. It should carry a removable lid to which a second stiff handle is attached so that after the can has been immersed in the liquid, the lid can be removed allowing the container to fill.

A.2.2.2 *A stout steel stirring rod,* suitable for stirring contents of a container.

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 (1)	Number of containers to be selected (2)
Up to 10	2
11 to 20	3
21 to 50	5
51 to 100	7

A.3.2 The containers should 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 TEST SAMPLE

A sufficient quantity of material should be drawn from each container selected as in A.3.1 using an appropriate method given in A.4.1 and A.4.2.

A.4.1 Preservatives in powder form

Three samples should be taken from the top, middle and bottom positions of each container selected for testing, using the sampling apparatus described in A.2.1. The contents should be transferred to an air-tight sample container of about 2 kg capacity and mixed well.

The entire sample should be transferred to a clean dry surface. A conical heap should be formed by placing 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. The cone should then be turned over to form a new cone. This operation should be carried out three times.

The third cone obtained accordingly should be flattened 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. This operation should be carried out until the flattened heap is of uniform thickness and diameter, and the centre coincides with the centre of the original cone.

The heap should then be divided into four portions (quartering) along two diameters which intersect at right angles, using a suitable divider. One pair of opposite quarters should be made into a heap, rejecting the remainder.

Coning and quartering should be carried out three times as described above until about 200 g of gross sample remains. The utmost care should be taken to reduce to a minimum the moisture picked up during the sampling, mixing and reducing processes.

If necessary, the whole sample should be ground to pass a test sieve of 2.00 mm aperture size and mixed well. The sample should be transferred to an air-tight container until ready for analysis.

A.4.2 Preservatives in paste or liquid form

The preservative in each container should be mixed thoroughly before samples are taken. Any settled material should be displaced from the bottom of the containers using the stirring rod. The containers should be shaken and rolled (preferably mechanically) to effect complete homogenisation of the contents. The containers should then be reopened and examined for uniformity by probing with the steel rod. This mixing procedure should be continued until the contents are completely homogenous.

Three samples should then be taken from each container from the top, middle and bottom positions of the containers using the sampling can. The three samples from each container should be poured into a clean glass or plastic container and mixed together to form a composite sample.

A.5 NUMBER OF TESTS

A.5.1 The containers 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 4.1 to 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, when tested as in A.5.2 satisfy the relevant requirements.

APPENDIX B
ANALYSIS OF THE PRESERVATIVE BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

B.1 PRINCIPLE

The preservative is treated with a mixture of dilute sulfuric acid and hydrogen peroxide solution. The resulting solution after addition of sodium sulfate solution is analysed using atomic absorption spectrometry.

B.2 REAGENTS

B.2.1 *Sulfuric acid,* 2.5 mol/l solution.
Cautiously add, with stirring and cooling, 280 ml of sulfuric acid (rel.den.=1.84) to 1600 ml of water. Cool and dilute to 2 liters with water.

B.2.2 *Hydrogen peroxide,* 300 g/l solution.

B.2.3 *Sodium sulfate,* 30 g/l solution.
Dissolve 30 g of anhydrous sodium sulfate in water and dilute to 1 litre with water.

B.2.4 *Sulfuric acid 0.5 moles per litre/ sodium sulfate 3 g/l solution.*
Dilute 200 ml of sulfuric acid solution **(B.2.1)** and 100 ml of 30 g/l sodium sulfate solution **(B.2.3)** to 1 litre with water and mix.

B.2.5 *Standard solution (One millilitre is equivalent to 500 µg of copper, 1000 µg of chromium and 1000 µg of arsenic)*
Dissolve 0.9825 g of copper sulfate pentahydrate ($CuSO_4.5H_2O$) in a little water and transfer the solution to a 500-ml one-mark volumetric flask. Dissolve 1.4135 g of anhydrous potassium dichromate ($K_2Cr_2O_7$) in a little water, add 50 ml of sulfuric acid solution **(B.2.1)** and 10 ml of hydrogen peroxide solution **(B.2.2)**, boil until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer the mixture to the one-mark volumetric flask containing the copper sulfate solution. Dissolve 0.6600 g of arsenic trioxide (As_2O_3) by boiling it in a solution containing 50 ml of sulfuric acid solution **(B.2.1)**, 10 ml of hydrogen peroxide solution **(B.2.2)** and 75 ml of water. Continue boiling until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer it to the one-mark volumetric flask. Add 50 ml of sodium sulfate solution **(B.2.3)** to the volumetric flask, dilute to the mark with water and mix.

B.3 APPARATUS

B.3.1 *Volumetric glassware*

B.3.2 *Atomic absorption spectrometer,* together with suitable sources of resonance radiation for copper, chromium and arsenic, for instance hollow-cathode lamps.

B.4 PROCEDURE

B.4.1 Instrument settings and operation

The instrument settings and operating conditions for the determination of copper, chromium and arsenic shall be as recommended in the instrument users' manual. Copper is determined in a fuel-lean air/acetylene flame at 324.8 nm, chromium in a fuel-rich air/acetylene flame or a fuel-lean nitrous oxide/acetylene flame at 357.9 nm or 429.0 nm, and arsenic in an argon/hydrogen or nitrous oxide/acetylene flame at 193.7 nm or 197.2 nm.

NOTE

It is essential that particular care be exercised when igniting the argon/hydrogen flame and that the instructions given in the instrument users' manual be followed precisely.

B.4.2 Preparation of calibration solutions

Transfer portions of 0.5 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml, and 7 ml of the standard solution (B.2.5) to a series of 100-ml one-mark volumetric flasks, dilute to the mark with the sulfuric acid/sodium sulfate solution (B.2.4) and mix.

One millilitre portions of the solutions so constituted contain 2.5 µg, 5 µg, 10 µg, 15 µg, 20 µg, 25 µg, 30 µg and 35 µg, respectively, of copper and 5 µg, 10 µg, 20 µg, 30 µg, 40 µg, 50 µg, 60 µg and 70 µg, respectively, of chromium and of arsenic.

NOTE

The range of the set of calibration solutions is given as a guide; being a function of the instrumental sensitivity, it can be varied. If varied, it is for the analyst to adjust the concentration of the material to be analysed in the test solution for presentation to the instrument in order to obtain optimum conditions, after checking that the possible interferences remain corrected and that others do not appear.

B.4.3 Analysis

B.4.3.1 Pipette a suitable quantity of preservative solution (see Note) to a 250-ml conical flask, add 40 ml of sulfuric acid solution **(B.2.1)** and with caution, 8 ml of hydrogen peroxide solution **(B.2.2)**. Boil the contents of the flask until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool the flask to room temperature and transfer its contents to a 200-ml one-mark volumetric flask. Add 20 ml of sodium sulfate solution **(B.2.3)**, dilute to the mark with water and mix to give the test solution.

NOTE

For 20 g/l solutions of preservative, 3 ml is suitable.

B.4.3.2 Using the operating conditions suitable for the instrument, aspirate the sulfuric acid/sodium sulfate solution **(B.2.4)** to obtain the blank absorbance, followed by the calibration solutions and then the test solution(s). Check the calibration solutions after the last test solution has been run.

NOTE

If a number of samples are to be analysed it may be advisable to check the instrument stability by bracketing each sample with appropriate standard solutions.

Plot calibration graphs of µg/ml of copper, chromium, and arsenic against absorbance. Determine the contents of copper, chromium, and arsenic in the test solutions by comparing the absorbance readings with the calibration graphs.

B.5 CALCULATION

B.5.1 Concentration, of copper or chromium or arsenic, in g/100 ml of test solution $= \frac{c_1}{50V}$

where,

c_1 is the concentration of the appropriate metal, in µg/ml of the test solution **(B.4.3.1)**; and

V is the volume, in ml, of the preservative solution taken in **B.4.3.1.**

B.5.2 To express the results in terms of copper sulfate (as $CuSO_4.5H_2O$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentrations of the respective metals by the following factors :

C_{copper} by 3.93;
$C_{chromium}$ by 2.87 and ;
$C_{arsenic}$ by 1.77

B.5.3 Copper or chromium or arsenic content in the preservative, per cent by mass $= \frac{c_2}{c_3} \times 1000$

where,

c_2 is the concentration of the ingredient obtained in B.5.2, in g/100 ml of the test solution, and
c_3 is the concentration, in g/l, of the preservative solution used in B.4.3.1.

APPENDIX C
ANALYSIS OF PRESERVATIVE USING COLORIMETRIC METHOD

C.1 PRINCIPLE

Preservatives are treated with a mixture of dilute sulfuric acid and hydrogen peroxide solution. Copper and chromium, in portions of the resulting solution, are allowed to react with zinc dibenzyldithiocarbamate and diphenylcarbazide respectively. The arsenic is converted to arsine, and reacted with silver diethyldithiocarbamate. The concentrations of the resulting coloured complexes are measured spectrometrically.

C.2 REAGENTS

C.2.1 *Sulfuric acid,* 2.5 mol/l solution.
Cautiously add, with stirring and cooling, 140 ml of concentrated sulfuric acid (rel. den. = 1.84) to 800 ml of water. Cool and dilute to one litre with water.

C.2.2 *Hydrogen peroxide,* 300 g/l solution.

C.2.3 *Sulfuric acid,* 0.5 mol/l solution.
Cautiously add, with stirring, 28 ml of concentrated sulfuric acid (rel. den. = 1.84) to 900 ml of water. Cool and dilute to one litre with water.

C.2.4 *Zinc dibenzyldithiocarbamate,* 1 g/l solution.
Dissolve 0.5 g of zinc dibenzyldithiocarbamate
$\{(C_6H_5.CH_2)_2NCS.S\}]_2Zn]$ in 500 ml of carbon tetrachloride.

C.2.5 *Standard solution,* prepared as prescribed in B.2.5.

C.2.6 *Standard copper solution* (One millilitre is equivalent to 2 μg of copper)
Pipette 10 ml of standard solution (C.2.5) to a 250-ml one-mark volumetric flask, make up to the mark with water and mix. Pipette 10 ml of this solution to a 100-ml one-mark volumetric flask, add 20 ml of sulfuric acid solution (C.2.1), make up to the mark with water and mix.

C.2.7 *Standard chromium solution* (One millilitre is equivalent to 100 μg of chromium.)
Pipette 10 ml of standard solution (C.2.5) to a 100-ml one-mark volumetric flask, dilute to the mark with water and mix.

C.2.8 *Standard arsenic solution* (One millilitre is equivalent to 1 μg of arsenic)
Pipette 10 ml of standard solution (C.2.5) to a 1000-ml one-mark volumetric flask, dilute to the mark with water and mix. Pipette 10 ml of this solution to a 100-ml one-mark volumetric flask, dilute to the mark with water and mix.

C.2.9 *Potassium permanganate,* 0.02 mol/l solution.
Dissolve 0.32 g of potassium permanganate in water and dilute to 100 ml with water.

C.2.10 *Sodium azide,* 50 g/l solution.
Dissolve 5 g of sodium azide (NaN_3) in water and dilute to 100 ml with water.

NOTE

Prepare the solution in a fume cupboard as sodium azide liberates poisonous fumes in contact with water or acids. Prevent contact with skin and eyes.

C.2.11 *1,5-diphenylcarbazide,* 10 g/l solution.
Dissolve 0.5 g of 1,5-diphenylcarbazide [$(C_6H_5NH.NH_2)_2CO$] in 40 ml of acetone containing 3 drops of sulfuric acid solution **(C.2.1),** and dilute to 50 ml with acetone. (It is essential that this solution be freshly prepared.)

C.2.12 *Silver diethyldithiocarbamate,* 5 g/l solution.
Dissolve 1.0 g of silver diethyldithiocarbamate [$(C_2H_5)_2N.CS.SAg$] in 200 ml of pyridine (C_5H_5N). Store in a dark glass bottle, preferably in the dark as the solution is not very stable, and renew the solution after two months.

C.2.13 *Potassium iodide,* 150 g/l solution.
Dissolve 15 g of potassium iodide (KI) in water and dilute to 100 ml with water.

C.2.14 *Tin (II) chloride,* 336 g/l solution.
Dissolve 40 g of tin (II) chloride dihydrate ($SnCl_2.2H_2O$) in 100 ml of concentrated hydrochloric acid solution (rel. den. = 1.18).

C.2.15 *Zinc,* 20-30 mesh or granulated, arsenic-free.

C.2.16 *Hydrochloric acid,* concentrated (rel. den = 1.18), arsenic-free.

C.2.17 *Lead (II) acetate,* 100 g/l solution.
Dissolve 10 g of lead (II) acetate trihydrate [$(CH_3COO)_2Pb.3H_2O$] in water and dilute to 100 ml with water.

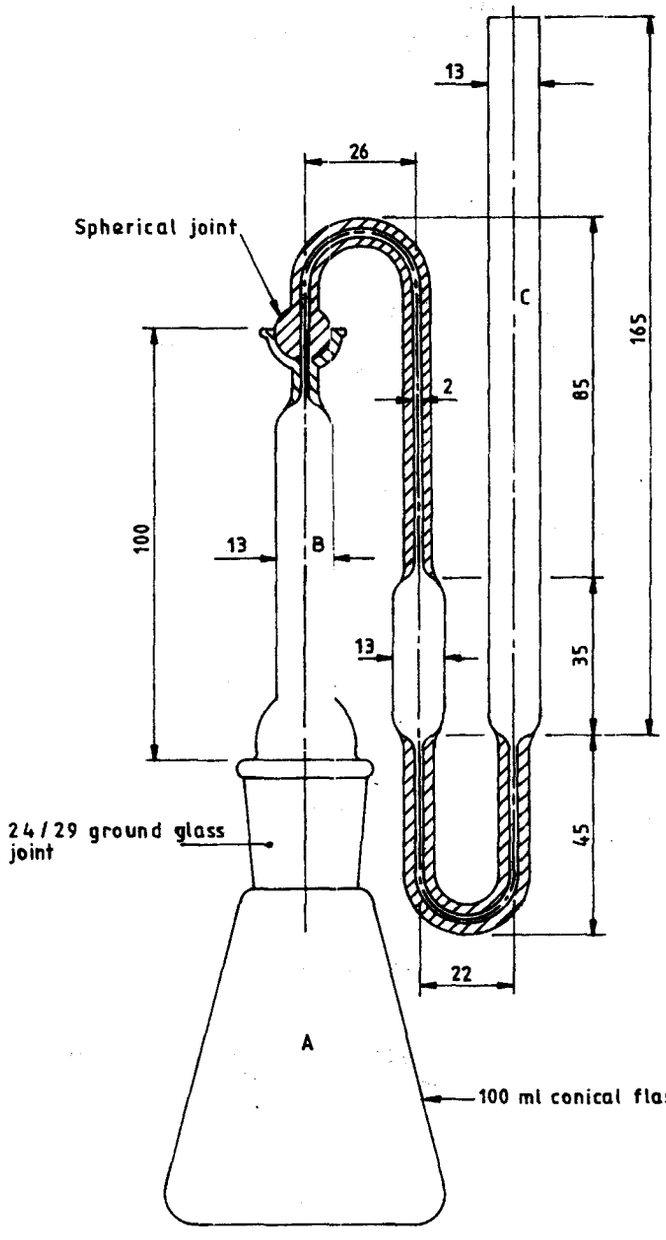
C.3 APPARATUS

C.3.1 *Volumetric glassware*

C.3.2 *Spectrometer or photoelectric absorptiometer,* suitable for the measurement of absorption at wavelengths of 435 nm and 540 nm.

C.3.3 *Apparatus for arsenic determination,* as given in Figure 2.

All dimensions in millimetres



A - Conical flask for evolution of arsine
B - Connecting tube to trap hydrogen sulfide
C - Absorption tube

FIGURE 2 - Apparatus for determination of arsenic

C.4 PROCEDURE

C.4.1 *Preparation of test solution*

Pipette a suitable quantity of preservative solution (see Note) to a 250-ml conical flask, add 50 ml of the sulfuric acid solution (C.2.1) and, with caution, 10 ml of hydrogen peroxide solution (C.2.2). Boil the contents of the flask until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, add 100 ml of water, cool the flask to room temperature and transfer the contents to a 250-ml one-mark volumetric flask. Dilute to the mark with water and mix to give the test solution.

NOTE

For 20 g/l solutions of preservative, 4 ml is suitable.

C.4.2 *Preparation of calibration graphs*

C.4.2.1 *Copper calibration*

Pipette, portions of 2 ml, 5 ml, 10 ml, 15 ml and 20 ml of the standard copper solution (C.2.6) to a series of 250-ml separating funnels. Proceed for each portion as detailed in C.4.3.1. The portions taken contain the equivalent of 4 µg, 10 µg, 20 µg, 30 µg and 40 µg respectively, of copper. Prepare the calibration graph by plotting the absorbances against micrograms of copper.

C.4.2.2 *Chromium calibration*

Pipette, portions of 0.1 ml, 0.2 ml, 0.4 ml, 0.6 ml, 0.8 ml, 1.0 ml, 1.2 ml, and 1.3 ml of the standard chromium solution (C.2.7) to a series of 25-ml beakers and dilute to 5 ml with water in each case. Add 5 ml of the sulfuric acid solution (C.2.3) to each beaker together with 0.5 ml of the potassium permanganate solution (C.2.9). Proceed for each portion from the appropriate point, as detailed in C.4.3.2. The portions taken contain the equivalent of 10 µg, 20 µg, 40 µg, 60 µg, 80 µg, 100 µg, 120 µg and 130 µg, respectively, of chromium. Prepare the calibration graph by plotting the absorbances against micrograms of chromium.

C.4.2.3 *Arsenic calibration*

Pipette, portions of 1 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the standard arsenic solution (C.2.8) to a series of 100-ml conical flasks (A in the figure) and dilute each to 22 ml with water. Proceed for each portion from the appropriate point, as detailed in C.4.3.3. The portions taken contain the equivalent of 1 µg, 2 µg, 4 µg, 6 µg, 8 µg and 10 µg, respectively, of arsenic. Prepare the calibration graph by plotting the absorbances against micrograms of arsenic.

C.4.3 **Analysis**

C.4.3.1 *For copper*

Pipette, 1 ml of the test solution (C.4.1) to a 250-ml separating funnel, dilute to 100 ml with sulfuric acid solution (C.2.3) and swirl to mix. Pipette 10 ml of the zinc dibenzyldithiocarbamate solution (C.2.4) and shake the separating funnel for 90 seconds. Allow the phases to separate. Run off the carbon tetrachloride layer through a dry 70 mm filter paper, for instance Whatman No. 1 filter paper, discarding the first runnings, into a suitable cell. Measure the absorbance of the yellow complex against a reagent blank prepared in a similar way, at a wavelength of 435 nm. Determine the copper content of the solution, by comparing the absorbance value with the calibration graph (see C.4.2.1).

C.4.3.2 *For chromium*

Pipette, 1 ml of the test solution (C.4.1) to a 25-ml beaker and gently evaporate to a small volume, but do not evaporate off the sulfuric acid. Allow the solution to cool, then add 6 ml of water, 4 ml of the sulfuric acid solution (C.2.3), and 0.5 ml of the potassium permanganate solution (C.2.9). Heat on a steam bath for 20 minutes, adding potassium permanganate solution dropwise as necessary, to maintain a slight excess. Then carefully add sodium azide solution (C.2.10) to the hot solution at the rate of about 1 drop every 10 seconds swirling after each addition. Continue until the solution is clear. It is important to avoid an excess of sodium azide in the solution. Remove the beaker immediately from the steam bath and cool it to room temperature. Transfer the solution to a 100-ml one-mark volumetric flask containing 15 ml of the sulfuric acid solution (C.2.3) and 60 ml of water. Swirl to mix. Add 2 ml of the 1,5-diphenylcarbazide solution (C.2.11), dilute to the mark with water and mix. Measure the absorbance of the violet-red complex against a reagent blank, prepared in a similar way, in suitable cells at a wavelength of 540 nm. Determine the chromium content of the solution, by comparing the absorbance value with the calibration graph (see C.4.2.2).

C.4.3.3 *For arsenic*

Pipette, 5 ml of the test solution (C.4.1) to a 200-ml one-mark volumetric flask and dilute to the mark with water. Pipette, 5 ml of this solution to the 100-ml conical flask (A in Figure 2), make up to 22 ml with water, and add 5 ml of hydrochloric acid solution (C.2.16), 2 ml of potassium iodide solution (C.2.13), and 8 drops of tin (II) chloride solution (C.2.14).

NOTE

The production of arsine should be carried out in a fume cupboard.

Swirl to mix and allow to stand for 15 minutes. Impregnate the glass wool or cotton wool in the connecting Tube B (see Figure 2) with lead (II) acetate solution (C.2.17) and charge the absorption Tube C with 4 ml of silver diethyldithiocarbamate solution (C.2.12). Add 5.0 g of zinc (C.2.15) to the solution in the 100-ml conical flask and immediately connect the flask to the tube assembly. The evolution of arsine is 99% complete in 30 minutes and virtually complete in about 40 minutes. Check that the volume of solution in the absorption tube is still 4 ml, if necessary, add sufficient pyridine to make up to 4 ml and mix. Measure the absorbance of the violet-red complex against a reagent blank, prepared in a similar way, in suitable cells at a wavelength of 540 nm. Determine the arsenic content of the solution, by comparing the absorbance value with the calibration graph (see C.4.2.3).

C.5 CALCULATION

C.5.1 Concentration of copper or chromium or arsenic, in g/100 ml of test solution is given by :

$$\frac{c_1}{40\ V} \quad \text{for copper}$$

$$\frac{c_1}{40\ V} \quad \text{for chromium}$$

$$\frac{c_1}{5\ V} \quad \text{for arsenic}$$

where,

c_1 is the appropriate metal content, in µg of the solution submitted to the spectrometer; and

V is the volume, in ml, of the preservative solution taken in C.4.1

C.5.2 To express the results in terms of copper sulfate (as $CuSO_4.5H_2O$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentrations of the respective metals by the following factors :

C_{copper} by 3.93;
$C_{chromium}$ by 2.87; and
$C_{arsenic}$ by 1.77.

C.5.3 Copper or chromium or arsenic content in preservative, per cent by mass $= \frac{c_2}{c_3} \times 1000$

where,

c_2 is the concentration of the ingredient obtained in C.5.2, in g/100 ml of the test solution; and

c_3 is the concentration, in g/l of the preservative solution used in C.4.1.

APPENDIX D
DETERMINATION OF pH

D.1 APPARATUS

D.1.1 *Sintered glass filter,* of pore size index P 40.

D.1.2 *Volumetric flask,* one-mark, 500-ml capacity.

D.1.3 *pH meter.*

D.2 PROCEDURE

D.2.1 Weigh, to the nearest milligram, a mass of the preservative equivalent to 10 ± 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 (D.1.1), into the volumetric flask (D.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 Appendix E)}

D.2.2 Determine the pH of the test solution at 27 ± 1 °C using a pH meter.

APPENDIX E
DETERMINATION OF INSOLUBLE MATTER CONTENT

E.1 APPARATUS

E.1.1 *Sintered glass filter,* of pore size index P 40.

NOTE

The sintered glass filter should be clean enough.

E.1.2 *Oven,* capable of maintaining at 105 ± 5 °C.

E.2 PROCEDURE

E.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 D.2.1, or if the pH has already been determined, use the residue on the filter.

E.2.2 Transfer the sintered filter containing the residue to the drying oven (E.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.

E.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.

APPENDIX F
FIRST-AID TREATMENT

Remove contaminated clothing. If skin is contaminated, wash immediately with plenty of running water.

In case of contact with eyes flush with plenty of water.

In the event of accidental ingestion, body contamination or feeling unwell, seek medical advice immediately.

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