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Capsulated dental silicate and silico-phosphate filling materials

Produits d'obturation dentaire en capsules, à base de silicates et de silicophosphates

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3851 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in August 1975.

It has been approved by the member bodies of the following countries:

| | | |
|---------------------|-----------------------|----------------|
| Australia | Iran | Sweden |
| Brazil | Mexico | Switzerland |
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| Germany | Romania | |
| India | South Africa, Rep. of | |

The member body of the following country expressed disapproval of the document on technical grounds:

U.S.A.

Capsulated dental silicate and silico-phosphate filling materials

0 INTRODUCTION

This International Standard has been prepared taking into account the contents of both ISO/R 1565, *Dental silicate cement*, and ISO 3824, *Dental silico-phosphate cements*. The significant difference is the specific coverage of the capsulated packaging, which has resulted in a number of basic changes in the test procedures and requirements. These changes include the specimen size, the reference point for setting time, and the use of the consistency test.

The silicate and silico-phosphate materials covered by this specification are those intended for use as dental fillings. For convenience of expression, however, and in accordance with common usage, the term "cement" has been used throughout the text.

At the present time it is capsulated silicate cements, rather than silico-phosphate, which are of more significance in terms of clinical usage, and the former have therefore been used as the main basis of the specification limits and tests. In the event of this situation changing, a more emphasis could be given to the silico-phosphate group of materials.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies requirements for dental silicate and silico-phosphate filling materials, presented as two separate preweighed components contained in a capsule and intended for mechanical mixing. The powder component is a finely ground alumina silicate glass, together with metal oxides, such as zinc oxide in the case of silico-phosphate cements, and the liquid is a solution of orthophosphoric acid and metal phosphates.

2 REFERENCE

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*.

3 REQUIREMENTS

3.1 Material

The cement shall consist of a powder and a liquid which, when mixed according to the manufacturer's instructions, will set to a condition suitable for its intended dental use.

3.2 Components

3.2.1 Liquid

The liquid shall form no obvious deposits or filaments on the inside of its container.

3.2.2 Powder

The powder shall be free from extraneous materials; when coloured, the pigment shall be uniformly dispersed throughout the powder.

3.3 Unset cement

3.3.1 Mixing

The cement, when mixed mechanically as directed in the manufacturer's instructions, shall be of a homogeneous, smooth consistency. In the event of the contents of a capsule being found not to comply with this requirement during mixing for the test methods described in the specification, then a further capsule shall be taken for examination. Such defective capsules shall not comprise more than 2 % of a test sample (i.e. 1 in 50).

3.3.2 Variation of cement consistency

The consistency of the mixed cement from any nine capsules, when determined in accordance with 5.3, shall not deviate more than 10 % from the mean of the ten tested.

3.4 Colour of set cement

The colour of the set cement of any shade selected at random from capsules of a test sample shall match the manufacturer's shade guide within the limits of professional acceptance. The sample shall be immersed in water for at least five days before comparison with the manufacturer's shade guide.

3.5 Physical properties

The time of setting, compressive strength, opacity¹⁾, solubility and disintegration shall be as specified in the table, when determined in accordance with the relevant test method given in clause 5.

1) The opacity requirement is not applicable to silico-phosphate cements.

3.6 Toxicity

3.6.1 General

The mixed cement, when used in accordance with the manufacturer's instructions, shall neither cause prolonged damage to oral tissues nor have any adverse systemic effect [see also 3.7 e)].

3.6.2 Arsenic content

The total arsenic content of the cement shall be as specified in the table, when tested in the manner described in clause 5.

3.7 Manufacturer's instructions

Adequate information on the following shall be included in the manufacturer's instructions :

- the method of bringing about physical contact between the powder and liquid;
- the method and timing of mechanical mixing;
- the setting time of the cement;
- the time normally available for manipulation and placement of the cement, when mixed in accordance with the manufacturer's instructions;
- if the cement can cause pulpal irritation, information on the use of a base/liner or other recommended protective measures;
- the minimum time at which finishing may be commenced.
- the minimum net volume of mixed cement in one capsule.

4 SAMPLING

A test sample shall comprise two retail packages of 50 or more capsules from different manufacturing batches.

A single retail package of 50 capsules is sufficient for the specified tests, but the second package is necessary in the event of repeat testing for the consistency test (5.3) being required.

5 TEST METHODS

5.1 Preparation of test specimens (General)

5.1.1 Ambient temperature

Unless stated otherwise all equipment shall be maintained and the preparation of all test specimens shall be carried out at 23 ± 1 °C.

5.1.2 Mixing

Use the mechanical mixing technique outlined in the manufacturer's instructions, subject to calibration of the timing device against a stop-watch prior to each series of tests. Include any necessary correction in the subsequent timing of the capsule mixing for specimen preparation.

5.2 Inspection procedures

Visual inspection should be used in determining compliance with 3.3.1, 3.4 and clause 6.

5.3 Consistency

5.3.1 Apparatus

5.3.1.1 Loading device of the type illustrated in figure 1, or an equivalent means whereby a load of 15,0 kg mass may be applied vertically onto the cement specimen; the surfaces of the upper and lower anvils of the loading device must be parallel.

5.3.1.2 Two flat glass plates 40 mm square and approximately 3 mm thick.

5.3.1.3 Measuring device to deliver 0,075 ml of mixed cement in the form of a cylinder 6,0 mm high and 4,0 mm in diameter; a suitable device would be a glass tube and a PTFE plunger.

5.3.2 Procedure

After mixing in accordance with the manufacturer's instructions, collect and load the cement into the measuring device. Deliver 0,075 ml of the mixed cement, preferably as an upright cylinder, onto the centre of one of the glass

TABLE — Requirements for capsulated dental silicate and silico-phosphate cements

| Time of setting* at 37 °C | | Strength in compression | Opacity** | | Solubility | Arsenic content |
|------------------------------|------|-------------------------------------|-------------------|------|--|-----------------|
| minutes | | MN/m ² | C _{0,70} | | Max. | mg/kg (ppm) |
| Min. | Max. | Min. | Min. | Max. | | Max. |
| 2 | 5 | 170 (1 750 kgf/cm ²) | 0,35 | 0,55 | 9,0 mg phosphate calculated as P ₂ O ₅ per gram of cement | 2 |

* It should be noted that the time of setting is determined from the completion of mixing (see 5.3).

** Silicate cements only.

plates, which is resting on the lower anvil of the loading device. If it is not possible to deliver all of the cement from the measuring device in a single operation, take the residue with the tip of a clean spatula and place on the centre of the other glass plate. Position both glass plates relative to each other, without pressure, in such a way that any cement on the second glass plate contacts centrally the bulk of the cement on the first glass plate.

Sixty seconds after the end of mixing, press the cement out between the two glass plates with a load of 15 kg applied in a direction perpendicular to the lower glass plate, avoiding any rotation of the glass plates.

After the cement has set, measure the major and minor diameter of the cement disc to an accuracy of 0,5 mm and then calculate the mean.

Carry out this test with ten capsules and determine the mean of the 10 results rounded off to the nearest 0,5 mm.

5.3.3 Test results

A sample shall be considered satisfactory if the individual results obtained from any nine capsules do not deviate more than $\pm 10\%$ from the mean of the total ten determinations.

If it is not the case, repeat the test, using the second test sample, and pay special attention to the correct manipulation of the capsules. If this sample is also unsatisfactory, then the product shall be considered unsatisfactory.

5.4 Time of setting¹⁾

5.4.1 Apparatus

5.4.1.1 Oven or cabinet in which the specimen may be maintained at a temperature of $37 \pm 1^\circ\text{C}$; no humidity control is necessary provided that the relative humidity is not allowed to fall below 30 % RH.

5.4.1.2 Indentor needle, weighing 400 ± 2 g and having a flat end of $1,0 \pm 0,1$ mm diameter; the needle tip shall be cylindrical for a distance of approximately 5,0 mm; the needle end shall be plane and at right angles to the axis of the rod.

5.4.1.3 Brass rectangular type moulds as illustrated in figure 2.

5.4.1.4 Metal block of minimum dimensions 8 mm \times 20 mm \times 10 mm, either as part of 5.4.1.1 or 5.4.1.2 or as separate item.

5.4.2 Procedure

Place the metal block and indentor needle in the oven at $37 \pm 1^\circ\text{C}$.

Place the brass rectangular mould, conditioned to $23 \pm 1^\circ\text{C}$, on a piece of aluminium foil of convenient size and fill to a level surface with cement.

One minute after the completion of mixing, place the filled mould on the metal block, in the oven.

One and a half minutes after completing the mix, carefully lower the indentor needle vertically onto the surface of the cement and allow to remain there for 5 s. Repeat this at 30 s intervals until near the expected time of setting, at which stage it should be carried out at 15 s intervals. Maintain the needle in a clean condition by cleaning, if necessary, between indentations.

Record the setting time as the period of time which elapses from the completion of mixing to the time when the needle fails to make a perceptible circular indentation on the surface of the cement, when viewed under a hand lens of low magnification.

Use the mean of three such tests in determining the setting time.

5.5 Strength in compression

5.5.1 Apparatus

5.5.1.1 Oven or cabinet maintained at a temperature of $37 \pm 1^\circ\text{C}$; no humidity control is necessary provided that the relative humidity is not allowed to fall below 30 % RH.

5.5.1.2 Five split moulds and plates, such as shown in figure 3, with internal dimensions 6 mm high and 4 mm diameter²⁾, made of stainless steel or other suitable material that will not be attacked or corroded by the cement.

5.5.1.3 Five individual screw clamps such as shown in figure 3.

5.5.1.4 Compressive strength testing apparatus with a cross-head speed of $0,75 \pm 0,25$ mm/min.

1) It should be noted that the time of setting determined by this test method is that from the completion of mixing, and NOT the more usual total setting time, as applied to hand-mixed cements, where the time is measured from first contact between the cement components.

2) The difference in behaviour between a 6 mm \times 4 mm specimen and one of 8 mm \times 4 mm, i.e. 1,5 : 1 and 2 : 1, has been found to be of no significance in relation to the test procedure described.

5.5.2 Preparation of test specimens

Bring the moulds, screw clamps and top and bottom plates to $23 \pm 1^\circ\text{C}$. After mixing in accordance with the manufacturer's instructions, pack the cement, to a slight excess, into the split mould within 1 min of the completion of mixing.

NOTE — In order to consolidate the cement and avoid trapping air, it is advisable to convey the largest convenient portions of mixed cement to the mould and apply to one side with a suitable instrument. Fill the mould to excess in this manner and then place on the bottom plate with some pressure.

Remove any bulk extruded cement, place the top metal plate in position and squeeze together. Put the mould and plates in the clamp and screw tightly together. Not later than 2 min after the completion of mixing, transfer the whole assembly to an oven at $37 \pm 1^\circ\text{C}$.

One hour after the completion of mixing, remove the plates and surface the ends of the specimen plane at right angles to its long axis, using a small amount of $45\ \mu\text{m}$ silicon carbide powder or similar abrasive, mixed with water on a flat glass plate. Alternatively an equivalent grade of abrasive-coated paper, and water, may be used. Keep both ends of the specimen wet during the grinding and rotate it about a quarter turn every few strokes.

Remove the specimen from the mould immediately after surfacing and rapidly check for air-voids or chipped edges. Discard any specimens where these defects are found.

NOTE — To facilitate the removal of the hardened cement specimen, the internal surface of the mould may be evenly coated, prior to filling, with a thin (3 %) solution of micro-crystalline or paraffin wax in pure toluene. Alternatively a thin film of silicone grease or PTFE dry film lubricant may be used.

Immerse the specimen in distilled water and maintain at $37 \pm 1^\circ\text{C}$ for 23 h.

Prepare at least five test specimens.

5.5.3 Procedure

Twenty-four hours after the completion of mixing, determine the compressive strength of the test specimens in the following manner, using the compressive strength testing apparatus.

Place the specimen with the flat ends between the platens of the testing apparatus so that the load is applied in the long axis of the specimen.

Record the maximum load applied when the specimen fractures, and calculate the compressive strength C in newtons per square millimetre ($\text{N}/\text{mm}^2 \equiv \text{MN}/\text{m}^2$), using the following formula :

$$C = \frac{4P}{\pi d^2}$$

where

P is the maximum applied load, in newtons;

d is the diameter of the specimens, in millimetres.

Determine the value for the compressive strength as the mean of three or more from the total of five determinations, and report the result to three significant figures. Discard any values for individual specimens which fall more than 15 % below the mean of the five, and report the mean of the remaining specimens. Repeat the whole test if more than two of the specimens have to be discarded.

5.6 Opacity¹⁾

5.6.1 Apparatus

5.6.1.1 Oven or cabinet maintained at a temperature of $37 \pm 1^\circ\text{C}$; no humidity control is necessary, provided that the relative humidity is not allowed to fall below 30 % RH.

5.6.1.2 Opal glass standards with $C_{0,70}$ values of 0,35 and 0,55 respectively.

5.6.1.3 Sheet of white waterproof material (approximately 110 mm × 40 mm) marked, along its entire length, with black stripes 2 mm wide and 3 mm apart.

5.6.1.4 Mould consisting of a split brass or stainless steel ring contained in a former as illustrated in figure 4; the height of the ring shall be $1,0 \pm 0,03$ mm and the internal diameter 10 mm.

5.6.1.5 Single or multiple spring clamp such as that shown in figure 5. Condition the spring clamp by placing in the oven at least 5 min before preparing the test specimen; do not remove until required.

NOTE — The contrast ratio $C_{0,70}$ used to represent the opacity is the ratio between the daylight apparent reflectance of the cement specimen when backed by a black backing, and the daylight apparent reflectance of 70 % relative to magnesium oxide (MgO).

1) Omit this sub-clause if the specification is being used for testing silico-phosphate cements.

5.6.2 Preparation of test specimen

Place the mould on a thin polyethylene or cellulose acetate sheet backed by a flat glass plate. Fill the split ring with cement mixed in accordance with the manufacturer's instructions from a popular light shade of powder. Cover with a further glass plate faced with a sheet of polyethylene or cellulose, and press firmly together.

Two minutes after the completion of mixing, place the mould and plates into the spring clamp which is in the oven at a temperature of $37 \pm 1^\circ\text{C}$.

After 1 h, remove the glass plates and polyethylene or cellulose acetate sheeting from the clamp and carefully separate the cement specimen from the ring. Store the specimen for 23 h in distilled water, or water of equal purity, maintained at $37 \pm 1^\circ\text{C}$.

5.6.3 Procedure

Make a comparison of the opacity of the cement specimen and the two opal glass standards by placing the specimen and the standards on the black and white striped background. Cover the cement specimen, the opal glass standards and the striped background with a thin film of distilled water while making the comparison.

If the translucency of the cement specimen is between those of the two standards or equal to either of them, it shall be considered to comply with this requirement.

Any photometric instrument may be used to make this comparison, provided that it can be proved to have an accuracy of within $\pm 0,02 C_{0,70}$.

5.7 Solubility

5.7.1 Apparatus

5.7.1.1 Oven or cabinet maintained at a temperature of $37 \pm 1^\circ\text{C}$; no humidity control is necessary provided that the relative humidity is not allowed to fall below 30 % RH.

5.7.1.2 Mould consisting of a split brass or stainless steel ring contained in a former as illustrated in figure 4; the height of the ring shall be $1,0 \pm 0,03$ mm and the internal diameter 10 mm.

5.7.1.3 Single or multiple spring clamp such as that shown in figure 5; condition the spring clamp by placing in the oven at least 5 min before preparing the test specimen; do not remove until required.

5.7.1.4 Platinum wire or waxed dental floss or equivalent non-corrodible material.

5.7.1.5 Two suitable wide-mouthed bottles of approximately 50 ml capacity, as illustrated in figure 6.

5.7.2 Reagents

Use reagents of nationally recognized analytical grade and distilled water or water of equal purity.

5.7.2.1 Phosphate standard. Prepare by dissolving 200 mg of anhydrous disodium hydrogen phosphate in 1 l of water. This will give a solution containing the equivalent of $100 \mu\text{g}$ of P_2O_5 per millilitre. Prepare a working standard containing $10 \mu\text{g}$ of P_2O_5 per millilitre by diluting 10 ml of this standard to 100 ml.

5.7.2.2 Reagent I. A 10 % solution of ammonium molybdate in normal ammonia (32 ml of concentrated ammonia solution, ρ 0,88 g/ml, in 500 ml of solution).

5.7.2.3 Reagent II. Sulphuric acid, 20 N.

5.7.2.4 Reagent III. A 4 % aqueous solution of ascorbic acid (it is essential that this solution be freshly prepared).

5.7.2.5 Reagent IV. Prepare by mixing carefully 40 ml of reagent I and 60 ml of reagent II; allow to cool, and add 100 ml of reagent III. It is essential that this solution be freshly prepared.

5.7.3 Preparation of test specimen

Place the mould on a thin polyethylene or cellulose acetate sheet backed by a flat glass plate.

Insert a convenient tared length of wire or dental floss¹⁾ through the split ring so that at least 4 mm projects into the ring. Fill the split ring with the contents of a single capsule mixed in accordance with the manufacturer's instructions. Cover with a further glass plate faced with a sheet of polyethylene or cellulose acetate and press firmly together.

Two minutes after the completion of mixing, place the mould and plates into the spring clamp which is in the oven at a temperature of $37 \pm 1^\circ\text{C}$.

After 1 h, remove the glass plates and polyethylene or cellulose acetate sheets from the clamp and carefully separate the cement disc and attached wire or dental floss from the split ring. Remove any surplus cement from the edge of the disc and lightly brush the surface to remove any loose material.

5.7.4 Preparation of test solution

Weigh the specimen. Immediately suspend the specimen in 20 ml of water contained in the plastics bottle, by means of the wire or dental floss. Ensure that the specimen does not touch the side of the bottle. Close the lid as tightly as possible and store for 23 h at $37 \pm 1^\circ\text{C}$.

1) In order to prevent breakage of the specimen while being removed from the split ring, it has been found advisable to splay the end of the dental floss.

5.7.5 Procedure

After 23 h, remove the specimen from the water and transfer the contents of the polyethylene bottle to a 100 ml flask. Dilute with water to the 100 ml calibration mark. Transfer 10 ml aliquot portions of this solution to 50 ml volumetric flasks, add 5 ml of reagent IV to each, then dilute the contents with water to the calibration marks and thoroughly mix. Treat 10 ml of the standard phosphate solution similarly by adding 5 ml of reagent IV and making the volume up to 50 ml in a volumetric flask. At the same time also prepare a blank. Allow these flasks to stand for 24 h and then compare the solutions at 650 nm in a suitable spectrophotometer. Carry out this determination in duplicate.

5.8 Arsenic content

The test method detailed in this sub-clause, or an instrumental method of comparable accuracy, shall be used in all cases to determine the total arsenic present in each shade of powder. If after carrying out this test the amount detected is judged to be near the permitted limit, then the test method specified in ISO 2590 shall be used to determine more precisely the exact amount present.

5.8.1 Apparatus

A wide-mouthed bottle of capacity about 120 ml is fitted with a rubber bung through which passes a glass tube. The latter, made from ordinary glass tubing, has a total length of 200 mm and an internal diameter of exactly 6,5 mm (external diameter about 8 mm). It is drawn out at one end to a diameter of about 1 mm, and a hole not less than 2 mm in diameter is blown in the side of the tube, near the constricted part. When the bung is inserted in the bottle containing 70 ml of liquid, the constricted end of the tube is above the surface of the liquid and the hole in the side is below the bottom of the bung. The upper end of the tube is cut off square, and is either slightly rounded or ground smooth.

Two rubber bungs (about 25 mm × 25 mm), each with a hole bored centrally and true, exactly 6,5 mm in diameter, are fitted with a rubber band or spring clip for holding them tightly together. Alternatively the two bungs may be replaced by any suitable contrivance satisfying the conditions described in the test procedure.

A suitable apparatus is shown in figure 7.

5.8.2 Standards and reagents

Use reagents of nationally recognized analytical quality and free from arsenic, and distilled water, or water of equal purity.

5.8.2.1 Arsenic solution, dilute

| | |
|------------------------------|--------|
| Strong arsenic solution : | 1 ml |
| Water, sufficient to make up | 250 ml |

Dilute arsenic solution shall be freshly prepared. 1 ml contains 0,004 mg of arsenic (As).

5.8.2.2 Arsenic solution, concentrated

| | |
|------------------------------|---------|
| Arsenic(III) oxide : | 0,132 g |
| Hydrochloric acid : | 50 ml |
| Water, sufficient to make up | 100 ml |

5.8.2.3 Lead acetate solution

A solution in carbon dioxide-free containing 10,0 g of lead acetate $[\text{CH}_3\text{COO}]_2\text{Pb} \cdot 3\text{H}_2\text{O}$ per 100 ml.

5.8.2.4 Mercury(II) chloride paper

Smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of mercury(II) chloride, pressed to remove superfluous solution, and dried at about 60 °C in the dark. The grade of the filter paper shall be such that the mass in grams per square metre is between 65 and 120; the thickness in millimetres of 400 papers shall be approximately equal, numerically, to the mass in grams per square metre.

NOTE — Mercury(II) chloride paper shall be stored in a stoppered bottle in the dark. Paper which has been exposed to sunlight or to the vapour of ammonia affords a lighter coloured stain or no stain at all when employed in the quantitative test for arsenic.

5.8.2.5 Hydrochloric acid, 10 N.

5.8.2.6 Potassium iodide.

5.8.2.7 Tin(II) chloride solution in hydrochloric acid (10 N), containing 33 g of tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) per 100 ml.

5.8.2.8 Stannated hydrochloric acid.

| | |
|-----------------------------|--------|
| Tin(II) chloride solution : | 1 ml |
| Hydrochloric acid : | 100 ml |

5.8.2.9 Zinc.

5.8.3 Standard stain

Prepare a solution by mixing 50 ml of water, 10 ml of stannated hydrochloric acid and 1 ml of diluted arsenic standard. The resulting solution, when treated as described in the test procedure, yields a stain on the mercury(II) chloride paper referred to as the standard stain.

5.8.4 Preparation of sample

Powder the set cement obtained by mixing ten capsules and sieve through a 75 µm (200 mesh) sieve. Disperse 2,0 g of the sieved powder in 50 ml of water and add 10 ml of stannated hydrochloric acid. Use this solution in the test for total arsenic.

5.8.5 Procedure

Lightly pack the glass tube with cotton or glass wool, previously moistened with lead acetate solution and dried, so that the upper surface of the wool is not less than

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25 mm below the top of the tube. Then insert the upper end of the tube into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm when the tube has a rounded-off end, or so that the ground end of the tube is flush with the larger end of the bung. Place a piece of mercury(II) chloride paper flat on the top of the bung, and then place the other bung over it and secure by means of the rubber band or spring clip, in such a manner that the borings of the two bungs (or the upper bung and the glass tube) meet to form a true tube of 6,5 mm diameter interrupted by a diaphragm of mercury(II) chloride paper.

Instead of this method of attaching the mercury(II) chloride paper, any other method may be used provided :

- 1) that the whole of the evolved gas passes through the paper;
- 2) that the portion of the paper in contact with the gas is a circle of 6,5 mm diameter;
- 3) that the paper is protected from sunlight during the test.

Place the solution to be examined, prepared as specified, in the wide-mouthed bottle, add 1 g of potassium iodide and 10 g of zinc, and quickly place the prepared glass tube in position. Allow the reaction to proceed for 40 min. Compare by daylight the yellow stain which is produced on the mercury(II) chloride paper if arsenic is present with the standard stain produced by operating in a similar manner with a known quantity of dilute arsenic solution. The standard stains used for comparison shall be freshly prepared (they fade on keeping).

By matching the depth of colour with the standard stain, it will be possible to estimate if the total arsenic content of the test sample approaches the specified limit of 2 mg/kg (2 ppm).

NOTES

- 1 The action may be accelerated by placing the apparatus on a warm surface, care being taken that the mercury(II) chloride paper remains quite dry throughout the test.
- 2 The most suitable temperature for carrying out the test is generally about 40 °C, but because the rate of evolution of the gas varies somewhat with different batches of zinc, the temperature may be adjusted to obtain a regular, but not violent, evolution of gas.
- 3 The tube must be washed with hydrochloric acid, rinsed with water, and dried between successive tests.

6 CAPSULATION, PACKAGING AND MARKING

6.1 Capsulation

6.1.1 Materials

The capsules shall be made of materials which adequately

protect their contents and have no adverse effect on the quality of the product.

6.1.2 Separation of components

The two components contained in the capsule shall be separated so that premature mixing does not take place under normal conditions of storage.

6.1.3 Design

When the manufacturer's instructions are followed, the method of capsulation shall permit the mixing of the components to be readily carried out.

6.2 Packaging

The capsules of cement powder and liquid shall be packed in a convenient container, marked in accordance with 5.3.

6.3 Marking of container

6.3.1 Name or trade mark of manufacturer

6.3.2 Description of contents

A description of the contents, as silicate or silico-phosphate, shall be marked on each container of capsules.

6.3.3 Batch number

Each container of capsules shall be marked with a serial number or code, which refers to the manufacturer's records and date of manufacture for that particular batch of cement powder and liquid.

6.3.4 Number of capsules

The number of capsules in a container shall be marked on the outside of the container.

6.3.5 Shade

Each container of capsules shall be marked to indicate the colour, or colours, of the capsule contents.

6.4 Instructions for use

A copy of the manufacturer's instructions shall accompany each container of capsules, unless a set of more than one container is intended for sale as a single unit, when only one copy of the instructions need accompany each set.