INTERNATIONAL STANDARD 1565

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Dental silicate cement (hand-mixed)

Ciments dentaires aux silicates (mélange manuel)

First edition — 1978-06-15

UDC 616.314 : 615.463

Ref. No. ISO 1565-1978 (E)

Descriptors : dentistry, dental materials, dental cements, materials specifications, tests, test equipment, marking.

Price based on 9 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 1565 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in October 1976.

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

Australia	India	Sweden
Austria	Ireland	Switzerland
Canada	Mexico	Thailand
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Norway	
Germany	South Africa, Rep. of	

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 1565-1970, of which it constitutes a technical revision.

Dental silicate cement (hand-mixed)

0 INTRODUCTION

This International Standard was first published by ISO in 1970 as an ISO Recommendation based on FDI Specification No. 7. In common with the other ISO Recommendations in this initial series on dental materials, ISO/R 1559 to ISO/R 1567, it was then the subject of a programme of revision to bring its contents up to date on the basis of technical data from both ISO/TC 106 and the Fédération Dentaire Internationale. The latter organization undertook the secretariat responsibilities of the working groups which prepared this International Standard.

Of the various changes introduced in this revision, most stem from the use of a much smaller test specimen than previously. The reason for using the small test specimen is to align the test methods for hand-mixed materials as closely as possible with those for capsulated materials. In general, this has merely necessitated some adjustments in technique to accommodate the smaller test specimen, but in some instances, the water-leachable material test in particular, more basic changes in the test method have been made.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies requirements for manually mixed dental silicate cements based on the hardening reaction between a glass powder, the principal constituent of which is an aluminosilicate, and aqueous solutions of phosphoric acid which may contain metal ions.

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 liquid which, when mixed according to the manufacturer's instructions, will set rapidly to a condition suitable for its intended use.

3.2 Components

3.2.1 *Liquid*

The liquid shall be free from obvious deposits or filaments on the inside of its container.

3.2.2 *Powder*

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

3.3 Unset cement

The cement, when mixed as directed in 5.1, shall be of uniform smooth consistency, completely mixed, and shall not evolve gases.

3.4 Set cement

After immersion in water for 5 days, the colour of any shade of the set cement, when viewed under water by natural daylight, shall match the manufacturer's shade guide within the limits of professional acceptance.

3.5 Performance requirements

The cement shall comply with the requirements specified in the table when tested according to the methods prescribed in clause 5.

3.6 Arsenic

The total arsenic content shall not exceed the limit specified in the table, when tested in the manner prescribed in clause 5.

3.7 Toxicity

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.

TABLE – **Performance requirements (at standard testing consistency)**

Net setting time* at 37 °C minutes		**Strength in compression** (24 h) MPa	**Opacity** $C_{0,70}$		**Water-leachable material** (24 h) mg P_2O_5/g	**Arsenic content** mg/kg (ppm)
min.	max.	min.	min.	max.	max.	max.
2	5	170	0,35	0,55	9,0	2

* The setting time is determined from the **completion** of mixing.

3.8 Manufacturer's instructions

Instructions for bringing about physical contact of the powder and the liquid, and for the mixing of these materials to form the cement, shall be supplied by the manufacturer. The following details shall be included :

a) the temperature, condition and type of the slab and spatula;

b) the recommended powder/liquid ratio;

c) the rate of incorporation of the powder;

d) the time of mixing;

e) the setting time;

f) the maximum satisfactory working time after the end of mixing;

g) a statement that, when clinical conditions warrant, a liner should be placed between the cement and the dentine.

4 SAMPLING AND SAMPLES

4.1 Procurement

The method of procurement shall be the subject of an agreement between the manufacturer and the testing authority and shall be recorded.

4.2 Samples

A sample drawn from one batch shall provide sufficient powder and liquid to complete all the prescribed tests.

5 TEST METHODS

5.1 Preparation of test specimens

5.1.1 *Ambient conditions*

Unless stated otherwise, the preparation of all specimens shall be carried out at 23 ± 1 °C and at a relative humidity of 50 ± 5 %.

5.1.2 *Components*

All tests shall be carried out on specimens prepared from samples of the powder and liquid complying with 3.2.1 and 3.2.2.

5.1.3 *Apparatus for mixing*

5.1.3.1 Glass slab, approximately 150 mm long × 75 mm wide × 20 mm thick.

5.1.3.2 Spatula, inert to the cement.

All apparatus used for mixing and for testing shall be kept clean, dry and free from hardened particles of cement.

5.1.4 *Method of mixing*

Place known amounts of powder and liquid on the glass slab, divide the powder into approximately two halves and then divide one half into two quarters.

Commence mixing the components by incorporating the half portion of the powder into the liquid in the first 15 s. Follow this by incorporating the quarter portions each at intervals of approximately 15 s, making sure that each portion is thoroughly mixed before introducing the next portion. Spatulate the whole mass with reasonable pressure for a further 15 s using a small area of the slab.

The total mixing time shall be 1 min. No particles of powder and no unused liquid shall remain on the slab when the mix is completed. The final mix shall appear to be homogeneous.

5.2 Inspection requirements

Visual inspection shall be used in determining compliance with 3.2.1, 3.2.2, 3.3, 3.4 and clause 6.

5.3 Determination of powder/liquid ratio for standard testing consistency

5.3.1 *Apparatus*

5.3.1.1 Loading device of the type illustrated in figure 1, or an equivalent means whereby a force of 147 N (15 kgf) may be applied vertically on to the cement.

In figure 1, the anvil which is attached to the bottom of the rod carrying the load shall be horizontal and parallel to the base and shall incorporate a device for holding the larger glass plate (see 5.3.1.2) in contact with its surface. The second, smaller, glass plate shall be held on the base by guides to prevent movement or rotation when the load is applied. The load shall be capable of being applied smoothly and in such a manner that no rotational motion occurs. The two glass plates shall be capable of touching over their entire facing surfaces, without interference from guides, etc.

5.3.1.2 Two flat glass plates, approximately 50 mm and 40 mm square and approximately 5 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 may consist of a glass tube and a PTFE plunger.

5.3.1.4 Graduated syringe pipette having an accuracy of ± 0,001 ml.

5.3.2 *Procedure*

Carefully weigh out a trial amount of powder (300 to 450 mg) to an accuracy of 1 mg and transfer it to the glass mixing slab. Deliver 0,100 ml of liquid from the syringe pipette close to the powder.

After mixing in accordance with 5.1.4, 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 the lower glass plate, which is resting on the lower anvil of the loading device. If it is not possible to deliver all 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, gently press the cement out between the two glass plates with the force of 147 N (15 kgf) applied in a direction perpendicular to the lower glass plate.

After the cement has set, measure the major and minor diameters of the cement disc with an accuracy of 0,5 mm and calculate the mean. If the two measurements differ by more than 1 mm discard the result and repeat the test.

Make trial mixes of varying powder/liquid ratios until the mean diameter calculated from the major and minor diameter measured is 23 ± 1 mm. Check this result twice.

The powder/liquid ratio which gives the required mix consistency, called the "standard testing consistency", shall be used in the preparation of all test specimens for tests carried out in accordance with this International Standard.

5.4 Net setting time

The setting time determined by this test method is measured from the completion of mixing, and **not** the more usual total setting time, where the time is measured from the first contact between the cement components.

5.4.1 *Apparatus*

5.4.1.1 Oven or cabinet in which the specimen may be maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

5.4.1.2 Indentor of mass 400 ± 5 g and having a flat end of diameter 1,0 ± 0,1 mm. 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 Metal mould, similar to that illustrated in figure 2.

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

5.4.1.5 Aluminium foil.

5.4.2 *Procedure*

Place the metal rectangular mould, conditioned to 23 ± 1 °C, on a piece of aluminium foil of convenient size and fill to a level surface with cement of standard testing consistency.

One minute after the completion of mixing, place the assembly containing a specimen on the metal block, which has been conditioned to 37 ± 1 °C, and replace in the oven. Ensure good contact between the mould, foil and metal block.

One and a half minutes after the completion of mixing, carefully lower the indentor vertically onto the surface of the cement and allow to remain there for 5 s. Repeat this at intervals until near the expected time of setting, at which stage reduce the intervals to 15 s. 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.

Take the mean of three such recorded values, rounded to the nearest 15 s, as the test result.

5.5 Compressive strength

5.5.1 *Apparatus*

5.5.1.1 Oven or cabinet maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

5.5.1.2 Split mould and **plates**, such as shown in figure 3, with internal dimensions 6 mm high and 4 mm diameter, made of stainless steel or other suitable material that will not be attacked or corroded by the cement.

5.5.1.3 Individual screw clamps.

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

5.5.2 *Preparation of test specimens*

Bring the moulds, top and bottom plates and the screw clamps to 23 ± 1 °C. After mixing to the standard testing consistency, 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 manually 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 the oven maintained at 37 ± 1 °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.

Grind the ends flat and remove any excess cement by drawing back and forth on a glass plate with a small amount of 350 mesh silicon carbide powder, maximum particle size 45 µm, mixed with water. Keep both ends of the specimen wet during the grinding and rotate about one quarter turn every few strokes.

Remove the specimen from the mould immediately after surfacing and check for air-voids or chipped edges. Discard any such defective specimens.

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 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 each acceptable specimen in distilled or deionized water and maintain at 37 ± 1 °C for 23 h.

Five specimens shall be prepared and tested.

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 a suitable apparatus with a cross-head speed of 0,75 ± 0,25 mm/min.

Place each 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 megapascals, using the formula

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

where

P is the maximum applied load, in newtons;

d is the diameter of the specimen, in millimetres.

If at least four of the five results obtained are below the minimum strength specified in the table, the material shall be deemed to have failed the test. If at least four of the five results are above the minimum strength specified in the table, the material shall be deemed to have passed the test. In other cases prepare a further 10 specimens and obtain the median result for all 15 specimens. Round this value to two significant figures and record as the compressive strength.

5.6 Translucency/Opacity

5.6.1 *Apparatus*

5.6.1.1 Oven or cabinet maintained at a temperature of 37 ± 1 °C, and a relative humidity of at least 30 %.

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

5.6.1.3 A 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 Moulds 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 ± 0,03 mm and the internal diameter 10 mm.

5.6.1.5 Individual screw clamps.

NOTE – The constrast 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 the specimen when backed by a white backing having a daylight apparent reflection of 70 % relative to magnesium oxide (MgO).

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 5.1.4 using a light shade of powder. Cover with a further plate faced with a sheet of polyethylene or cellulose acetate, press firmly together and clamp. The specimen shall be 1,00 ± 0,05 mm thick.

Two minutes after the completion of mixing, place the mould, plates and the screw clamp into the oven maintained at a temperature of 37 ± 1 °C, and at a relative humidity of at least 30 %.

After 1 h, remove the plates and polyethylene or cellulose sheets from the clamp and carefully separate the cement specimen from the ring. Store the specimen for 23 h in distilled or deionized water maintained at 37 ± 1 °C.

5.6.3 *Procedure*

Make a comparison of the translucency 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 or deionized 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 ± 0,02 $C_{0,70}$.

5.7 Water-leachable material

5.7.1 *Apparatus*

5.7.1.1 Oven or cabinet maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

5.7.1.2 Mould consisting of a split brass or stainless steel ring contained in a former or retaining plate similar to that illustrated in figure 4. The height of the ring shall be 1,0 ± 0,03 mm and the internal diameter 10 mm. The former or retaining plate shall ensure that excess cement does not expand the split ring beyond a diameter of 10 mm.

5.7.1.3 Individual screw clamps.

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

5.7.1.5 Two wide-mouthed polyethylene bottles of approximately 50 ml capacity, as illustrated in figure 5.

5.7.1.6 Spectrophotometer having a range including 650 nm, with cells (optional); or a suitable comparator with Nessler tubes.

5.7.2 *Reagents*

All reagents shall be of analytical grade. Unless stated otherwise, distilled or deionized water shall be used.

5.7.2.1 Phosphate standard solution. Dissolve 200 mg of anhydrous disodium hydrogen orthophosphate in 1 l of water. This will give a solution containing the equivalent of 100 µg/ml of P_2O_5.

Prepare a working standard solution containing 10 µg/ml of P_2O_5 by diluting 10 ml of this standard solution to 100 ml.

5.7.2.2 Reagent I. A 10 % solution of ammonium molybdate in 1 N ammonia solution (33 ml of concentrated ammonia solution, 15 N, 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 % solution of ascorbic acid (it is essential that this solution be freshly prepared).

5.7.2.5 Reagent IV. Mix 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 plate.

Insert a convenient tared length of wire or dental floss through the split ring so that at least 4 mm projects into the ring. Fill the split ring with cement mixed to the standard testing consistency.

Cover with a further plate faced with a sheet of polyethylene or cellulose acetate, press firmly together and apply the screw clamp.

Two minutes after the completion of mixing, place the mould, plates and the screw clamp into the oven maintained at 37 ± 1 °C, and a relative humidity of at least 30 %.

After 1 h, remove the 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 and immediately suspend it in 20 ml of water, contained in a polyethyelne 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 ± 1 °C.

5.7.5 *Procedure*

After 23 h, remove the specimen from the water and determine the amount of phosphate in solution by the following procedure.

Carry out this determination in duplicate.

Transfer the contents of each of the polyethylene bottles to a 200 ml flask and dilute to the calibration mark with water. Transfer 10 ml aliquot portions of these solutions to 50 ml volumetric flasks, and add 5 ml of reagent IV to each, the contents then being diluted to the calibration marks and thoroughly mixed. 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.

If no spectrophotometer is available, the sample solution may be compared against a suitable standard; 9 ml of the working standard solution (5.7.2.1) approximates to the specification limit if the cement disc is of 0,2 g mass. Standard Nessler procedures should be adopted, but where any result is questionable, the spectrophotometer method shall be used.

NOTE — Nessler equipment used in the United States of America differs from that used in Europe.

5.7.6 *Expression of results*

The amount of water-leachable material, expressed as P_2O_5 eluted in milligrams per gram of the specimen, is given by the formula

$$\frac{A_1 - A_3}{A_2 - A_3} \times \frac{2}{m}$$

where

A_1 is the absorbance of the test solution;

A_2 is the absorbance of the standard phosphate solution;

A_3 is the absorbance of the blank solution;

m is the mass, in grams, of the specimen.

NOTES

1 The absorbance, A_2, of the standard phosphate solution measured in a 1 cm cell at 650 nm is normally about 0,260.

2 This formula differs from that used in ISO 1566.

5.8 Arsenic content

5.8.1 *Preparation of sample*

Powder the set cement and sieve through a 75 μm (200 mesh) sieve. Disperse 2 g of the sieved powder in 30 ml of water and add 10 ml of hydrochloric acid, 38 % (*m/m*) (ρ 1,19 g/ml). Use this solution in the test for total arsenic content.

5.8.2 *Procedure*

The total arsenic content may be determined using any recognized analytical method of adequate sensitivity.

If the result of such a determination shows the total arsenic content to be near the limit specified in the table, then a further determination shall be carried out using the procedure described in ISO 2590. The result obtained using ISO 2590 shall then be taken as the test result.

6 PACKAGING AND MARKING

6.1 Packaging

The components shall be supplied in securely sealed containers,[1] made from materials which do not react with or permit contamination of the contents.

6.2 Instructions for use

Instructions (detailed in 3.8) for proportioning powder and liquid and the recommended method for mixing shall accompany each package.

6.3 Marking of containers

Each container shall be clearly marked with the following particulars :

a) the name and/or trade mark of the manufacturer, and the class of cement;

b) the shade of the powder according to the manufacturer's shade guide;

c) the minimum net mass, in grams, of the powder and the minimum net volume, in millilitres, of the liquid;

d) a serial number or code number (optional) to identify each batch or lot, together with the actual date of manufacture (obligatory);

e) the number of this International Standard, i.e. ISO 1565.

BIBLIOGRAPHY

WILSON, A. D., and BATCHELOR, R. F. "Initial solubility and disintegration of dental silicate cements — A test with miniature specimens". *Brit. Dent. J.* **130**, 143-146 (1971).

1) For the purpose of this International Standard, the container shall be considered as the immediate wrapping of the component.

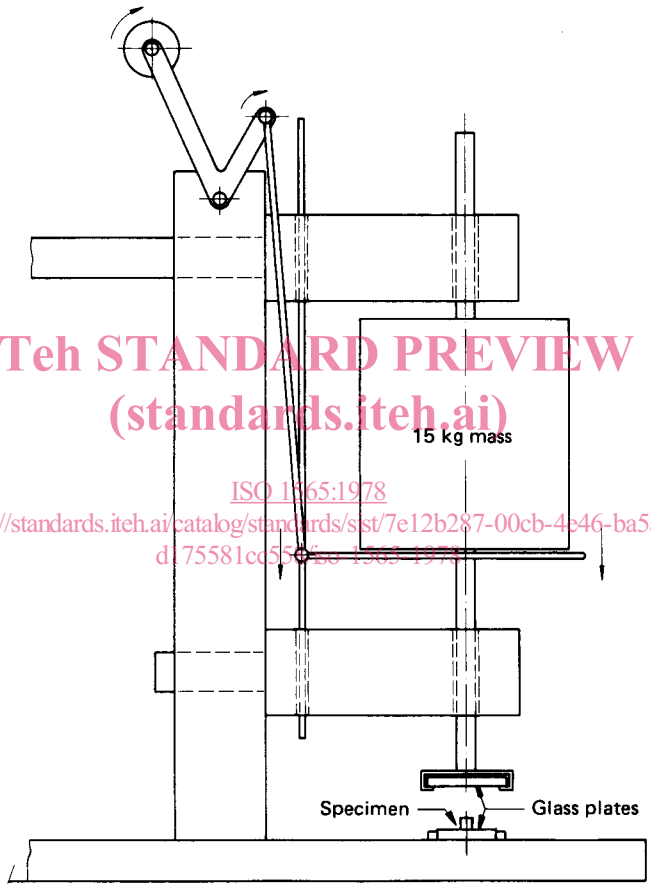

FIGURE 1 — **Loading device for measuring consistency**