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Sodium and potassium silicates for industrial use — Preparation of solution of products not easily soluble in boiling water and determination of matter insoluble in water

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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 2122 was drawn up by Technical Committee ISO/TC 47, *Chemistry.*

It was approved in March 1971 by the Member Bodies of the following countries:

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Poland	U.S.A.
Hungary	Portugal	U.S.S.R.

No Member Body expressed disapproval of the document.

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Sodium and potassium silicates for industrial use — Preparation of solution of products not easily soluble in boiling water and determination of matter insoluble in water

1 SCOPE

This International Standard specifies a method for dissolving sodium or potassium silicates that are not easily soluble in boiling water at atmospheric pressure, and also a method for the determination of matter insoluble in water.

NOTE — When the method described in this International Standard is used, the masses of sample laid down in ISO/R 1690 and ISO/R 1691 must be replaced by taking corresponding quantities of the solution, and the expression of the results must be modified accordingly.

2 FIELD OF APPLICATION

The method is applicable to the treatment of products corresponding to the general formula $M_2O.\ xSiO_2$, where the values of x lie between :

1.5 and 3.5 in the case of sodium silicates;

1.9 and 3.9 in the case of potassium silicates.

2.1 Special case

Preparation of solution of products corresponding to the general formula $M_2O.\ xSiO_2$, where the values of x are greater than the values shown above.

Because of the difficulty encountered in dissolving fine particles of these products, the sample is first sieved to remove these fines. The homogeneity of the fused material is considered to be sufficient to allow this size segregation.

3 REFERENCES

ISO/R 1686, *Sodium and potassium silicates for industrial use — Samples and methods of test — General.*

ISO/R 1690, *Sodium and potassium silicates for industrial use — Determination of silica content — Gravimetric method by insolubilization.*

ISO/R 1691, *Sodium and potassium silicates for industrial use — Determination of carbon dioxide content expressed as sodium or potassium carbonate — Gas-volumetric method.*

4 PRINCIPLE

Dissolution of the test portion in carbon dioxide-free water, by treatment in an autoclave.

Filtration and weighing of the insoluble matter on a tared filter crucible.

Dilution of the filtrate to a given volume, from which aliquot samples are taken in order to carry out the different determinations.

5 REAGENTS

5.1 Carbon dioxide-free water

This can be obtained either by boiling for 10 min, followed by cooling, with protection from atmospheric carbon dioxide by means of a soda lime tube, or by bubbling nitrogen or carbon dioxide-free air through distilled or demineralized water for approximately 10 min, and storage under protection from atmospheric carbon dioxide.

5.2 Indicator paper, covering neutral range (pH 6 to 8).

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Stainless steel autoclave, approximately 300 ml effective capacity allowing a pressure of the order of 600 kN/m^2 (i.e. 6 bars) to be reached. (See the Figure, for an example of a suitable autoclave.)

6.2 Filter crucible, with sintered disk of porosity between 15 and 40 μm.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0.1 g, 100 g of the laboratory sample (see ISO/R 1686) in the form of grains not exceeding 90 μm in size.

A different quantity may be taken, if using an apparatus of suitable capacity, provided that the ratio of product to water is maintained.

7.2 Preparation of solution

Place the test portion (7.1) in the autoclave (6.1), and add 200 ml of the water (5.1).

Heat the autoclave, immersed in an oil bath, at a temperature between 135 and 160 °C for 3 h in the case of sodium silicate, and at 150 °C (maximum) for 90 min in the case of potassium silicate. Measure the temperatures in the oil bath. Allow to cool to ambient temperature.

7.3 Determination of matter insoluble in water

Empty the autoclave. Rinse inside with water (5.1). Filter together under vacuum the solution and rinsing water on the filter crucible (6.2), previously tared to the nearest 0.1 mg after drying for 1 h at between 110 and 120 °C and cooling in a desiccator.

Wash the insoluble matter with the water (5.1) until the pH of the filtrate, measured with the aid of the indicator paper (5.2), is not greater than 7.

Dry the filter crucible and its contents for 1 h at a temperature between 110 and 120 °C, and reweigh the whole to the nearest 0.1 mg, after cooling in a desiccator to room temperature.

7.4 Preparation of the sample solution

Transfer the filtered solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water (5.1) and mix.

This solution, which should be used in carrying out the various determinations, contains approximately 0.1 g of sample per millilitre.

8 EXPRESSION OF RESULTS

Water insoluble matter is given, as a percentage by mass, by the formula :

$$(m_2 - m_3) \times \frac{100}{m_1}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the filter crucible containing the insoluble matter;

m_3 is the mass, in grams, of the filter crucible alone.

9 SPECIAL CASE

Preparation of solution of products corresponding to the general formula $M_2O.\ xSiO_2$, where the values of x are greater than 3.5 in the case of sodium silicates and 3.9 in the case of potassium silicates.

9.1 Principle

See section 4, the filter crucible, however, being replaced by filter paper.

9.2 Reagents

See section 5.

9.3 Apparatus

Ordinary laboratory apparatus and

9.3.1 Stainless steel autoclave, approximately 150 ml effective capacity, capable of being heated to 150 °C (the internal pressure at this temperature is of the order of 480 kN/m^2, i.e. 4.8 bars).

9.3.2 Ashless filter papers, medium texture, 125 mm diameter, resistant to the action of silicates.

9.4 Procedure

9.4.1 *Test portion*

Grind 100 to 200 g of the laboratory sample (see ISO/R 1686). Sift the material through two superimposed sieves of 710 µm and 180 µm mesh respectively, and regrind if necessary until the whole of the sample has passed through the 710 µm mesh sieve.

Weigh, to the nearest 0.01 g, 30 g of the fraction of sample having a particle size between 180 and 710 µm.

The method described below involves the dissolution of 30 g of the sample, when using the autoclave (9.3.1). A different quantity may be taken, if using an apparatus of suitable capacity, provided that the ratio of product to water is maintained.

9.4.2 *Preparation of solution*

Place the test portion (9.4.1) in the autoclave (9.3.1), add 100 ml of water (5.1) in the case of sodium silicate or 75 ml of water in the case of potassium silicate, and close the apparatus.

Rotate the autoclave on a suitable device for 90 min in a thermostatically controlled bath set at about 150 °C, then take it out and allow it to cool to room temperature.

9.4.3 *Determination of matter insoluble in water*

Open the apparatus and add to the contents about 50 ml of water heated to about 70 °C. Mix, then filter on a Büchner funnel under vacuum through a filter paper (9.3.2) which has been previously dried for 1 h at between 110 and 120 °C, cooled in a desiccator to room temperature and weighed to the nearest 0.1 mg in a weighing bottle.

Wash the insoluble matter in water (5.1) until the pH of the filtrate, measured with the aid of the indicator paper (5.2), is not greater than 7.

Remove the filter paper from the funnel and weigh it again in the same weighing bottle to the nearest 0.1 mg after drying it for 1 h at between 110 and 120 °C and cooling in a desiccator to room temperature.

9.4.4 *Preparation of the sample solution*

Transfer the filtered solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water (5.1) and mix.

This solution, which serves for carrying out the various determinations, contains about 0.03 g of the sample per millilitre.

9.5 Expression of results

Use the formula of section 8, where

m_2 is the mass, in grams, of the weighing bottle containing the filter paper and the insoluble matter;

m_3 is the mass, in grams, of the weighing bottle containing the filter paper only.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

Dimensions in millimetres

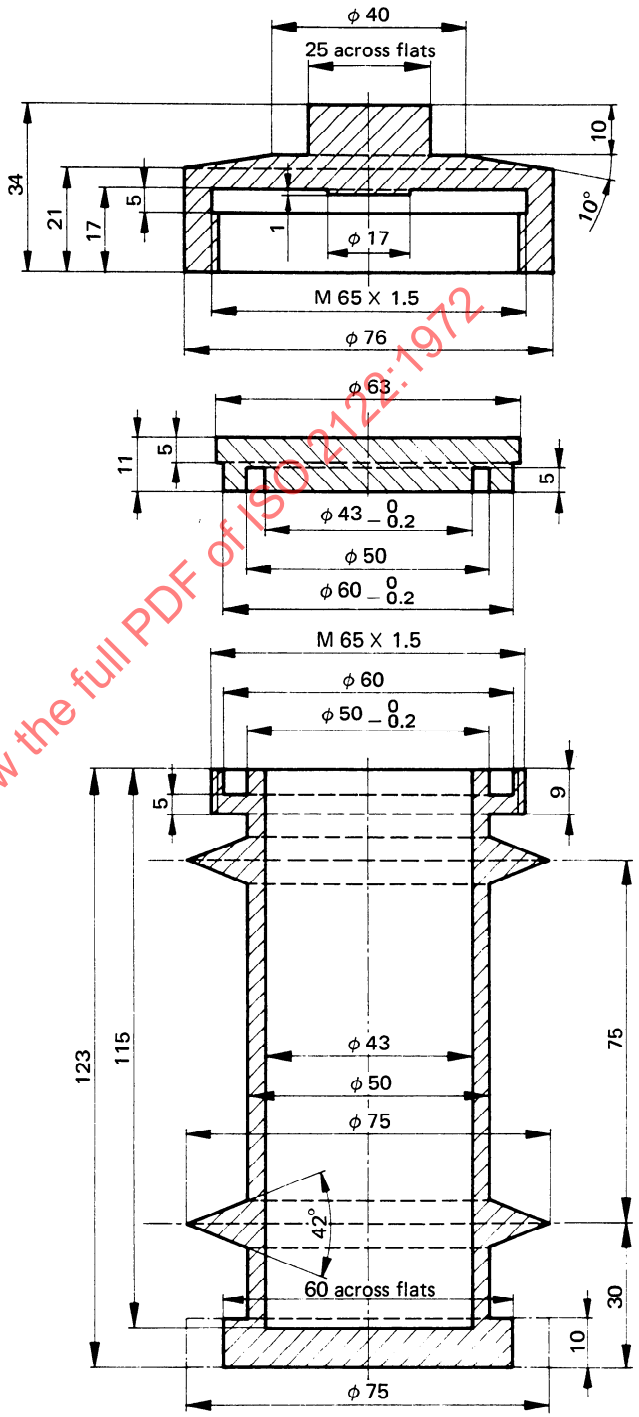

FIGURE — **Suitable autoclave for the preparation of an aqueous solution of silicates not easily soluble**

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