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**Cement — Test methods —  
Part 1:  
Analysis by wet chemistry**

*Ciments — Méthodes d'essai —*

*Partie 1: Analyse chimique par voie humide*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 29581-1 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

This first edition of ISO 29581 cancels and replaces ISO 680:1990, which has been technically revised as follows, based on comments received by the secretariat.

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- a) The scope of the analyses has been extended to include determination of chloride, carbon dioxide and alkali.
  - b) Calibration against internationally accepted reference materials is permitted.
  - c) The number of tests carried out when the analysis is part of a series subject to statistical control has been reduced to one.
  - d) A requirement for blank determinations has been included.
  - e) Limiting ranges have been set for masses, volumes and temperatures wherever these are significant.
  - f) The required accuracy of the balance is consistent with that of equipment traditionally used.
  - g) A specification for a laboratory oven has been included.
  - h) The calibration procedure for the standard silica solution has been simplified.
  - i) Additional indicators have been included for the visual determination of EDTA titrations.
  - j) An ignition temperature of  $(950 \pm 25)$  °C has been set for the determination of loss on ignition and the ignition of barium sulfate and insoluble residues.
  - k) Determination of sulfate before and after ignition in the determination of loss on ignition becomes the reference method when correcting for sulfide.
  - l) Determination of silica by the double evaporation method becomes the reference method.
  - m) Alternative ignition temperatures during the analysis for silica are permitted, where validated by the laboratory.

- n) An alternative method for the determination of chloride by potentiometric titration is included.
- o) An alternative method for the determination of acid-soluble alkali by atomic absorption spectroscopy is included.
- p) XRF and other instrumental methods, such as differential thermal analysis for determination of carbon dioxide, atomic absorption spectroscopy, etc. may be used as alternative methods provided they are calibrated against the reference methods, or against internationally accepted reference materials.

NOTE Analytical methods utilizing x-ray fluorescence (XRF) were considered during this revision but no published standardized method was considered sufficiently comprehensive to be included. A new Part 2 to ISO 29581 is under development in order to prepare a method based on XRF.

ISO 29581 consists of the following parts, under the general title *Cement — Test methods*:

- *Part 1: Analysis by wet chemistry*
- *Part 2: Analysis by x-ray fluorescence*

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# Cement — Test methods —

## Part 1: Analysis by wet chemistry

### 1 Scope

This part of ISO 29581 specifies the methods for the analysis of cement by wet chemistry.

This part of ISO 29581 gives the reference methods and, in certain cases, an alternative method that can be considered to be equivalent. In the case of a dispute, only the reference methods are used.

It is permitted to use other methods provided they are calibrated, either against the reference methods or against internationally accepted reference materials, in order to demonstrate their equivalence. In case of dispute, only the reference methods are used.

This part of ISO 29581 describes methods that apply principally to cements, but which can also be applied to their constituent materials. They can also be applied to other materials, the standards for which call up these methods.

Standard specifications state which methods are used.

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### 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 835, *Laboratory glassware — Graduated pipettes*

### 3 General requirements for testing

#### 3.1 Number of tests

Analysis of a cement can require the determination of a number of its chemical properties. For each determination, one or more tests shall be carried out in which the number of measurements taken shall be as specified in the relevant clause of this part of ISO 29581.

Where the analysis is one of a series subject to statistical control, determination of each chemical property by a single test shall be the minimum required.

Where the analysis is not part of a series subject to statistical control, the number of tests for determination of each chemical property shall be two (see also 3.3).

In the case of a dispute, the number of tests for determination of each chemical property shall be two (see also 3.3).

## 3.2 Repeatability and reproducibility

### 3.2.1 General

Repeatability and reproducibility in this part of ISO 29581 are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s) in, for example, absolute percent, grams, etc., according to the property tested.

### 3.2.2 Repeatability

Precision under repeatability conditions applies where independent test results are obtained with the same method on identical test items (material) in the same laboratory by the same operator using the same equipment within short intervals of time.

### 3.2.3 Reproducibility

Precision under reproducibility conditions applies where test results are obtained with the same method on identical test items (material) in different laboratories with different operators using different equipment.

## 3.3 Expression of masses, volumes, factors and results

Express masses in grams to the nearest 0,000 1 g and volumes from burettes in millilitres to the nearest 0,05 ml.

Express the factors of solutions, given by the mean of three measurements, to three decimal places.

Express the results, where a single test result has been obtained, as a percentage generally to two decimal places.

Express the results, where two test results have been obtained, as the mean of the results, as a percentage generally to two decimal places.

If the two test results differ by more than twice the standard deviation of repeatability, repeat the test and take the mean of the two closest test results.

The results of all individual tests shall be recorded.

## 3.4 Ignition

Carry out ignition as follows.

Place the filter paper and its contents into a crucible which has been previously ignited and tared. Dry it, then incinerate slowly in an oxidizing atmosphere in order to avoid immediate flaming, while ensuring complete combustion. Ignite the crucible and its contents at the stated temperature, then allow to cool to the laboratory temperature in a desiccator. Weigh the crucible and its contents.

## 3.5 Determination of constant mass

Determine constant mass by making successive 15 min ignitions followed each time by cooling and then weighing. Constant mass is reached when the difference between two successive weighings is less than 0,000 5 g.



### 3.6 Check for absence of chloride ions — Silver nitrate test

After generally five to six washes of a precipitate, rinse the base of the filter stem with a few drops of water. Wash the filter paper and its contents with several millilitres of water and collect this in a test tube. Add several drops of silver nitrate solution (4.2.43). Check the absence of turbidity or precipitate in the solution. If present, continue washing while carrying out periodic checks until the silver nitrate test is negative.

### 3.7 Blank determinations

Carry out a blank determination without a sample, where relevant, following the same procedure and using the same amounts of reagents. Correct the results obtained for the analytical determination accordingly.

## 4 Reagents

### 4.1 General

Use only reagents of analytical quality. References to water mean distilled or de-ionized water having an electrical conductivity  $\leq 0,5$  mS/m.

Unless otherwise stated, percent means percent mass fraction.

Unless otherwise stated, the concentrated liquid reagents used in this part of ISO 29581 have the following densities,  $\rho$ , at 20 °C, expressed in grams per cubic centimetre:

|                   |              |                    |              |
|-------------------|--------------|--------------------|--------------|
| hydrochloric acid | 1,18 to 1,19 | acetic acid        | 1,05 to 1,06 |
| nitric acid       | 1,40 to 1,42 | phosphoric acid    | 1,71 to 1,75 |
| perchloric acid   | 1,60 to 1,67 | ammonium hydroxide | 0,88 to 0,91 |

The degree of dilution is always given as a volumetric sum, for example: dilute hydrochloric acid 1 + 2 means that 1 volume of concentrated hydrochloric acid is mixed with 2 volumes of water.

### 4.2 Products used

- 4.2.1 Hydrochloric acid (HCl), concentrated,
- 4.2.2 Hydrochloric acid, dilute, 1 + 1.
- 4.2.3 Hydrochloric acid, dilute, 1 + 2.
- 4.2.4 Hydrochloric acid, dilute, 1 + 3.
- 4.2.5 Hydrochloric acid, dilute, 1 + 9.
- 4.2.6 Hydrochloric acid, dilute, 1 + 11.
- 4.2.7 Hydrochloric acid, dilute, 1 + 19.
- 4.2.8 Hydrochloric acid, dilute, 1 + 99.

**4.2.9 Hydrochloric acid**, dilute, pH 1,60 + 0,05.

Prepare by adjusting the pH of 2 l of water to  $1,60 \pm 0,05$  by adding five or six drops of concentrated hydrochloric acid (HCl) (4.2.1). Control using the pH meter (5.18.1). Store the solution in a polyethylene container.

**4.2.10 Hydrofluoric acid**, (HF), concentrated, > 40 %.

**4.2.11 Hydrofluoric acid**, dilute, 1 + 3.

**4.2.12 Nitric acid** (HNO<sub>3</sub>), concentrated.

**4.2.13 Nitric acid**, dilute, 1 + 2.

**4.2.14 Nitric acid**, dilute, 1 + 100.

**4.2.15 Sulfuric acid**, concentrated, (> 98 %) (H<sub>2</sub>SO<sub>4</sub>).

**4.2.16 Sulfuric acid**, dilute, 1 + 1.

**4.2.17 Sulfuric acid**, dilute, 1 + 4.

**4.2.18 Perchloric acid** (HClO<sub>4</sub>), concentrated.

**4.2.19 Phosphoric acid** (H<sub>3</sub>PO<sub>4</sub>), concentrated.

**4.2.20 Phosphoric acid**, dilute, 1 + 19.

Store this solution in a polyethylene container.

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**4.2.21 Boric acid** (H<sub>3</sub>BO<sub>3</sub>).

**4.2.22 Acetic acid** (CH<sub>3</sub>COOH), concentrated.

**4.2.23 Aminoacetic acid** (NH<sub>2</sub>CH<sub>2</sub>COOH).

**4.2.24 Chromium** (Cr), metallic, in powder form.

**4.2.25 Ammonium hydroxide** (NH<sub>4</sub>OH), concentrated.

**4.2.26 Ammonium hydroxide**, dilute, 1 + 1.

**4.2.27 Ammonium hydroxide**, dilute, 1 + 10.

**4.2.28 Ammonium hydroxide**, dilute, 1 + 16.

**4.2.29 Sodium hydroxide** (NaOH).

**4.2.30 Sodium hydroxide solution**, 4 mol/l.

Dissolve 160 g of sodium hydroxide (NaOH) (4.2.29) in water and make up to 1 000 ml. Store in a polyethylene container.

**4.2.31 Sodium hydroxide solution**, 2 mol/l.

Dissolve 80 g of sodium hydroxide (NaOH) (4.2.29) in water and make up to 1 000 ml. Store in a polyethylene container.

**4.2.32 Ammonium chloride** ( $\text{NH}_4\text{Cl}$ ).

**4.2.33 Tin(II) chloride** ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ).

**4.2.34 Potassium iodate** ( $\text{KIO}_3$ ), dried to constant mass at  $(120 \pm 5)^\circ\text{C}$ .

**4.2.35 Potassium periodate** ( $\text{KIO}_4$ ).

**4.2.36 Sodium peroxide** ( $\text{Na}_2\text{O}_2$ ), in powder form.

**4.2.37 Sodium chloride** ( $\text{NaCl}$ ), dried to constant mass at  $(110 \pm 5)^\circ\text{C}$ .

**4.2.38 Potassium chloride** ( $\text{KCl}$ ), dried to constant mass at  $(110 \pm 5)^\circ\text{C}$ .

**4.2.39 Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ), dried to constant mass at  $(250 \pm 10)^\circ\text{C}$ .

**4.2.40 Sodium carbonate and sodium chloride**, mixture.

Mix 7 g of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) (4.2.39) with 1 g sodium chloride ( $\text{NaCl}$ ) (4.2.37).

**4.2.41 Barium chloride solution.**

Dissolve 120 g of barium chloride ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ) in water and make up to 1 000 ml.

**4.2.42 Silver nitrate** ( $\text{AgNO}_3$ ), dried to constant mass at  $(150 \pm 5)^\circ\text{C}$ .

**4.2.43 Silver nitrate solution.**

Dissolve 5 g of silver nitrate ( $\text{AgNO}_3$ ) (4.2.42) in water, add 10 ml of concentrated nitric acid ( $\text{HNO}_3$ ) (4.2.12) and make up to 1 000 ml with water.

**4.2.44 Silver nitrate solution**, 0,05 mol/l.

Dissolve  $(8,494\ 0 \pm 0,000\ 5)$  g of silver nitrate ( $\text{AgNO}_3$ ) (4.2.42) in water in a 1 000 ml volumetric flask and make up to the mark. Store in a brown glass container and protect from the light.

**4.2.45 Sodium carbonate solution.**

Dissolve 50 g of anhydrous sodium carbonate (4.2.39) in water and make up to 1 000 ml.

**4.2.46 Potassium hydroxide solution.**

Dissolve 250 g of potassium hydroxide ( $\text{KOH}$ ) in water and make up to 1 000 ml. Store in a polyethylene container.

**4.2.47 Ammoniacal zinc sulfate solution.**

Dissolve 50 g of zinc sulfate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 150 ml water and add 350 ml of concentrated ammonium hydroxide (4.2.25). Leave to stand for at least 24 h and filter.

**4.2.48 Lead acetate solution.** Dissolve approximately 0,2 g of lead acetate [ $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ ] in water and make up to 100 ml.

**4.2.49 Starch solution.**

To 1 g of starch (water soluble), add 1 g of potassium iodide (KI), dissolve in water and make up to 100 ml. Use within two weeks.

**4.2.50 Polyethylene oxide solution.**

Dissolve 0,25 g of polyethylene oxide  $(-\text{CH}_2-\text{CH}_2-\text{O}-)_n$  of average molecular mass 200 000 to 600 000, in 100 ml water while stirring vigorously. Use within two weeks.

**4.2.51 Boric acid solution, saturated.**

Dissolve approximately 50 g of boric acid ( $\text{H}_3\text{BO}_3$ ) in water and make up to 1 000 ml.

**4.2.52 Citric acid solution.**

Dissolve 10 g of citric acid ( $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$ ) in water and make up to 100 ml.

**4.2.53 Calcium carbonate ( $\text{CaCO}_3$ ), dried to constant mass at  $(200 \pm 10)^\circ\text{C}$  (of purity > 99,9 %).**

**4.2.54 Ammonium molybdate solution.**

Dissolve 10 g of ammonium molybdate  $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$  in water and make up to 100 ml. Store the solution in a polyethylene flask. Use within one week.

**4.2.55 Copper sulfate solution.**

Dissolve 0,45 g of copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water and make up to 50 ml in a volumetric flask.

**4.2.56 Ammonium acetate solution.**

Dissolve 250 g of ammonium acetate ( $\text{CH}_3\text{COONH}_4$ ) in water and make up to 1 000 ml.

**4.2.57 Triethanolamine  $[\text{N}(\text{CH}_2\text{CH}_2\text{OH})_3]$ , of purity > 99 %, diluted to 1 + 4 solution.**

**4.2.58 Reducing solution.**

Dissolve 1 g of tin(II) chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) (4.2.33) in water to which has been added 1 ml of concentrated hydrochloric acid (4.2.1). Make up to 100 ml with water. Use within one day.

**4.2.59 Buffer solution, pH 1,40.**

Dissolve  $(7,505 \pm 0,001)$  g of amino-acetic acid (4.2.23) and  $(5,850 \pm 0,001)$  g of sodium chloride (NaCl) (4.2.37) in water and make up to 1 000 ml. Dilute 300 ml of this solution to 1 000 ml with hydrochloric acid 1 + 99 (4.2.8).

**4.2.60 Standard potassium iodate solution, approximately 0,016 6 mol/l.**

Weigh, to  $\pm 0,000 5$  g,  $(3,6 \pm 0,1)$  g, of potassium iodate ( $\text{KIO}_3$ ) (4.2.34), record as  $m_1$ , and place in a 1 000 ml volumetric flask. Add 0,2 g of sodium hydroxide (4.2.29), 25 g of potassium iodide (KI), dissolve all the solids in freshly boiled and cooled water and make up to the mark using the same water.

Calculate the factor,  $F$ , of the potassium iodate solution from Equation (1):

$$F = \frac{m_1}{3,5668} \tag{1}$$

where  $m_1$  is the mass of the weighed portion of potassium iodate, expressed in grams.

**4.2.61 Sodium thiosulfate solution**, approximately 0,1 mol/l.

#### 4.2.61.1 Preparation

Dissolve (24,82 ± 0,01) g of sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O) in water and make up to 1 000 ml. Before each test series, determine the factor,  $f_S$ , of this solution from Equation (2).

#### 4.2.61.2 Standardization

**4.2.61.2.1** This standardization is carried out preferably using the standard potassium iodate solution (4.2.60).

For this standardization, pipette 20 ml of the standard potassium iodate solution (4.2.60) into a 500 ml conical flask and dilute with approximately 150 ml of water. Acidify with 25 ml of hydrochloric acid 1 + 1 (4.2.2) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution (4.2.61) to a pale yellow colour. Add 2 ml of the starch solution (4.2.49) and continue the titration until the colour changes from blue to colourless.

Calculate the factor,  $f_S$ , of the sodium thiosulfate solution from Equation (2):

$$f_S = \frac{20 \times 0,01667 \times 214,01 \times F}{3,5668 \times V_1} \quad (2)$$

$$= 20 \times \frac{F}{V_1}$$

where

$F$  is the factor of the standard potassium iodate solution (4.2.60);

$V_1$  is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration, expressed in millilitres;

3,566 8 is the mass of potassium iodate corresponding to a solution with exactly 0,016 67 mol/l of potassium iodate, expressed in grams;

214,01 is the relative molecular mass of KIO<sub>3</sub>, expressed in grams.

**4.2.61.2.2** The standardization may alternatively be carried out using a known quantity of potassium iodate.

For this standardization, weigh, to ± 0,000 5 g, (0,070 ± 0,005) g of potassium iodate (4.2.34), record as  $m_2$ , and place in a 500 ml conical flask. Dissolve in approximately 150 ml of water. Add about 1 g of potassium iodide, acidify with 25 ml of hydrochloric acid 1 + 1 (4.2.2) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution (4.2.61) until a pale yellow colour is obtained. Then add 2 ml of the starch solution (4.2.49) and titrate until the colour changes from blue to colourless.

Calculate the factor,  $f_S$ , of the sodium thiosulfate solution from Equation (3):

$$f_S = \frac{1000 \times m_2}{3,5668 \times V_2} = 280,3634 \times \frac{m_2}{V_2} \quad (3)$$

where

$m_2$  is the mass of potassium iodate, expressed in grams;

$V_2$  is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration, expressed in millilitres;

3,566 8 is the mass of potassium iodate corresponding to a solution with exactly 0,016 67 mol/l of potassium iodate, expressed in grams.

**4.2.62 Manganese**

**4.2.62.1 Anhydrous manganese sulfate.**

Dry hydrated manganese sulfate ( $MnSO_4 \cdot xH_2O$ ) to constant mass at  $(250 \pm 10) ^\circ C$ . The composition of the product obtained corresponds to the formula  $MnSO_4$ .

**4.2.62.2 Standard manganese solution.**

**4.2.62.2.1 Preparation**

Into a 1 000 ml volumetric flask, weigh, to  $\pm 0,000 5$  g,  $(2,75 \pm 0,05)$  g of anhydrous manganese sulfate, record as  $m_3$ , dissolve in water and make up to the mark. Calculate the concentration,  $c_{Mn(II)}$ , of manganese(II) ions of this solution, expressed in milligrams of  $Mn^{2+}$  per millilitre, from Equation (4):

$$c_{Mn(II)} = \frac{m_3}{2,748 5} \tag{4}$$

where  $m_3$  is the mass of anhydrous manganese sulfate, expressed in grams.

**4.2.62.2.2 Construction of the calibration curve**

Into each of two volumetric flasks, respectively, 500 ml (No. 1) and 1 000 ml (No. 2), pipette 20 ml of the standard manganese solution. Make up to the mark with water. Into each of three volumetric flasks, respectively, 200 ml (No. 3), 500 ml (No. 4) and 1 000 ml (No. 5), pipette 100 ml of the solution from flask No. 2 and make up to the mark with water.

Take 100 ml of each solution from flasks 1 to 5 and pipette each portion into a 400 ml beaker. Add 20 ml of concentrated nitric acid (4.2.12), 1,5 g of potassium periodate (4.2.35) and 10 ml of phosphoric acid (4.2.19), heat to boiling and boil gently for 30 min.

Allow to cool to room temperature and transfer the contents of each beaker to a 200 ml volumetric flask and make up to the mark with water. Measure the absorbance of the solutions using a photometer (5.10) at a wavelength of around 525 nm, against water [use one or more cells (5.11) of appropriate sizes]. Record the absorbance values to three decimal places.

For each cell optical length, construct a separate curve of the absorbance of these calibration solutions E1 to E5 as a function of the corresponding manganese concentrations in milligrams of Mn per 200 ml. The corresponding manganese concentrations are given in Table 1. They can be used as given if the content,  $c_{Mn(II)}$ , obtained in accordance with Equation (4) has the value 1,000 0. Otherwise, multiply the manganese concentrations in Table 1 by the value of  $c_{Mn(II)}$  calculated from Equation (4).

**Table 1 — Concentrations of manganese calibration solutions**

| Calibration solution   | E1  | E2  | E3  | E4  | E5  |
|--|-----|-----|-----|-----|-----|
| Concentration of manganese, expressed in milligrams of Mn per 200 ml | 4,0 | 2,0 | 1,0 | 0,4 | 0,2 |

**4.2.63 Standard silica solution.**

**4.2.63.1 Silica ( $SiO_2$ ), of purity  $> 99,9$  % after ignition to constant mass at  $(1 175 \pm 25) ^\circ C$ .**

**4.2.63.2 Basic solution.**

Weigh  $(0,200 0 \pm 0,000 5)$  g of freshly ignited silica (4.2.63.1), in a platinum crucible already containing  $(2,0 \pm 0,1)$  g of anhydrous sodium carbonate (4.2.39).

Heat the mixture and fuse it at a bright-red heat for at least 15 min. After cooling to room temperature, place the fused solid in a polyethylene beaker, dissolve it in water, then transfer the solution quantitatively to a 200 ml volumetric flask and make up to the mark with water.

Store the solution in a polyethylene container.

This solution contains 1 mg of SiO<sub>2</sub> per millilitre.

#### 4.2.63.3 Standard solution.

Pipette 5 ml of the basic solution into a 250 ml volumetric flask and make up to the mark with water. Store the solution in a polyethylene container. This solution contains 0,02 mg silica per millilitre. Use within one week.

#### 4.2.63.4 Compensating solutions.

Prepare the compensating solutions according to the procedure adopted in the determination of silica content (13.3 to 13.5) by dissolving the amounts of the reagents given in Table 2 in water and making up to 500 ml.

#### 4.2.63.5 Construction of the calibration curve

Add from a burette the volumes of the silica calibration solutions given in Table 3 into 100 ml polyethylene beakers each containing a magnetic stirrer bar. Add 20 ml of the compensating solution by pipette and make up to 40 ml with water from a burette. The volumes required for this are also given in Table 3. While stirring with a magnetic stirrer, add 15 drops of hydrofluoric acid 1 + 3 (4.2.11). Stir for at least 1 min. Then pipette 15 ml of the boric acid solution (4.2.51) into the solution.

Table 2 — Composition of the compensating solutions for a volume of 500 ml

| Component and unit                                | Precipitation by double evaporation (see 13.3) | Precipitation by polyethylene oxide (see 13.4) | Decomposition by HCl and NH <sub>4</sub> Cl (see 13.5) |
|---|--|--|--|
| HCl, concentrated, millilitres                    | 75   | 70   | 15   |
| H <sub>2</sub> SO <sub>4</sub> 1 + 1, millilitres | 1  | 1  | —  |
| HNO <sub>3</sub> , concentrated, millilitres      | —  | —  | 1  |
| Polyethylene oxide solution, millilitres          | —  | 5  | -  |
| NH <sub>4</sub> Cl, grams                         | —  | —  | 1  |
| Na <sub>2</sub> CO <sub>3</sub> , grams           | 1,75   | 1,75   | 1,75   |
| NaCl, grams                                       | 0,25   | 0,25   | 0,25   |
| Na <sub>2</sub> O <sub>2</sub> , grams            | 3  | 3  | —  |

Table 3 — Composition of the silica calibration solutions and their silica content

| Serial No.                                      | Blank | 1    | 2    | 3    | 4    |
|---|-------|------|------|------|------|
| Standard SiO <sub>2</sub> solution, millilitres | 0     | 2    | 5    | 10   | 20   |
| Water, millilitres                              | 20    | 18   | 15   | 10   | 0    |
| Silica content, mg SiO <sub>2</sub> /100 ml     | 0     | 0,04 | 0,10 | 0,20 | 0,40 |