A.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 (see **A.4.2.27**). Check the absence of turbidity or precipitate in the solution. If present, continue washing while caring out periodic checks until the silver nitrate test is negative.

A.4 Reagents

A.4.1 General

Use only reagents of analytical quality. References to water mean distilled or deionized water having an electrical conductivity of not more than 0.5 mS/m.

Unless otherwise stated, percent means percent mass fraction.

Unless otherwise stated, the concentrated reagents used in this Standard have the following densities (ρ) (g/cm³, 20 °C):

- 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
- aqueous ammonia 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.

A.4.2 Reagents used

- A.4.2.1 Hydrochloric acid (HCl)
- A.4.2.2 Hydrochloric acid (1+2)
- A.4.2.3 Hydrochloric acid (1+19)
- A.4.2.4 Hydrochloric acid (1+99)

A.4.2.5 Hydrochloric acid (**pH 1.60**±0.05) Add 5 to 6 drops of hydrochloric acid (**A.4.2.1**) to 2 L of water, and adjust its pH to 1.60 ± 0.05 using a pH meter (**A.5.16.1**). Store in a polyethylene bottle.

A.4.2.6 Hydrofluoric acid (HF) (>40%)

- A.4.2.7 Hydrofluoric acid (1+3)
- A.4.2.8 Nitric acid (HNO₃)
- A.4.2.9 Sulfuric acid (H₂SO₄) (>98 %)

- **A.4.2.10** Sulfuric acid (1+1)
- **A.4.2.11** Boric acid (H₃BO₃)
- A.4.2.12 Acetic acid (CH₃COOH)
- A.4.2.13 Aminoacetic acid (NH₂CH₂COOH)
- A.4.2.14 Aqueous ammonia (NH₄OH)
- A.4.2.15 Aqueous ammonia (1+1)
- **A.4.2.16** Aqueous ammonia (1+10)
- A.4.2.17 Sodium hydroxide (NaOH)

A.4.2.18 Sodium hydroxide solution (4 mol/L) Dissolve 160 g of sodium hydroxide (A.4.2.17) in water to make 1 000 ml. Store the solution in a polyethylene bottle.

A.4.2.19 Sodium hydroxide solution (2 mol/L) Dissolve 80 g of sodium hydroxide (A.4.2.17) in water to make 1 000 ml. Store the solution in a polyethylene bottle.

A.4.2.20 Ammonium chloride (NH₄Cl)

A.4.2.21 Tin (II) chloride dihydrate $(SnCl_2 \cdot 2H_2O)$

A.4.2.22 Sodium peroxide (Na₂O₂) Powder

A.4.2.23 Sodium chloride (NaCl) Dry at 110 °C±5 °C to a constant mass.

A.4.2.24 Sodium carbonate (Na_2CO_3) Dry at 250 °C ± 10 °C to a constant mass.

A.4.2.25 Sodium carbonate and sodium chloride, mixture Mix 7 g of sodium carbonate (A.4.2.24) and 1 g of sodium chloride (A.4.2.23).

A.4.2.26 Silver nitrate (AgNO₃) Dry at 150 $^{\circ}C \pm 5 ^{\circ}C$ to a constant mass.

A.4.2.27 Silver nitrate solution Dissolve 5 g of silver nitrate (A.4.2.26) in water, add 10 ml of nitric acid (A.4.2.8) and add water to make 1 000 ml.

A.4.2.28 Polyethylene oxide solution Dissolve 0.25 g of polyethylene oxide $[(-CH_2-CH_2-O-)_n]$ of average molecular mass 200 000 to 600 000 in 100 ml of water while stirring vigorously. Use this solution within two weeks.

A.4.2.29 Boric acid solution Dissolve approximately 50 g of boric acid (**A.4.2.11**) in water to make 1 000 ml.

A.4.2.30 Citric acid solution Dissolve 10 g of citric acid monohydrate $(C_6H_8O_7 \cdot H_2O)$ in water to make 100 ml.

A.4.2.31 Calcium carbonate $(CaCO_3)~(>99.9~\%)~$ Dry at 200 $^\circ\mathrm{C}\pm10~^\circ\mathrm{C}$ to a constant mass.

A.4.2.32 Ammonium molybdate solution Dissolve 10 g of hexaammonium heptamolybdate tetrahydrate $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$ in water to make 100 ml. Store the solution in a polyethylene bottle. Use within one week.

A.4.2.33 Copper sulfate solution Dissolve 0.45 g of copper sulfate (II) pentahydrate $(CuSO_4 \cdot 5H_2O)$ in water, transfer into a 50-ml volumetric flask, and make up to the mark with water and shake to mix.

A.4.2.34 Ammonium acetate solution Dissolve 250 g of ammonium acetate (CH_3COONH_4) in water to make 1 000 ml.

A.4.2.35 Triethanolamine $[N(CH_2CH_2OH)_3]$ solution (1+4) The purity of triethanolamine to be used is over 99 %.

A.4.2.36 Reducing solution Dissolve 1 g of tin (II) chloride dihydrate (**A.4.2.21**) in water to which 1 ml of hydrochloric acid (**A.4.2.1**) has been added. Make up to 100 ml with water. Use within one day.

A.4.2.37 Buffer solution (pH 1.40) Dissolve 7.505 g \pm 0.001 g of amino-acetic acid (A.4.2.13) and 5.850 g \pm 0.001 g of sodium chloride (A.4.2.23) in water to make 1 000 ml. Dilute 300 ml of this solution to 1 000 ml with hydrochloric acid (1+99) (A.4.2.4).

A.4.2.38 Silicon dioxide standard solution

A.4.2.38.1 Silicon dioxide (SiO₂) (>99.9 %)

Ignite at 1 175 °C ± 25 °C to a constant mass.

A.4.2.38.2 Standard stock solution

Weigh 0.200 0 $g\pm 0.0005$ g of freshly ignited silicon dioxide (**A.4.2.38.1**) in a platinum crucible already containing 2.0 $g\pm 0.1$ g of sodium carbonate (**A.4.2.24**).

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 bottle.

This solution contains 1 mg of silicon dioxide per millilitre.

A.4.2.38.3 Standard solution

Pipette 5 ml of the standard stock solution into a 250-ml volumetric flask, make up to the mark with water and shake to mix. Store the solution in a polyethylene bottle. This solution contains 0.02 mg silicon dioxide per millilitre. Use within one week.

A.4.2.38.4 Compensating solutions

Prepare the compensating solutions according to the procedure adopted in the determination of silica content (see **A.13.3** to **A.13.5**) by dissolving the amounts of the reagents given in table **A.1** in water and making up to 500 ml.

Reagent	Precipitation by double evaporation (A.13.3)	Precipitation by polyethylene oxide (A.13.4)	Decomposition by hydrochloric acid-ammonium chloride (A.13.5)	
Hydrochloric acid (ml)	75	70	15	
Sulfuric acid (1+1) (ml)	1	1	_	
Nitric acid (ml)	—	—	1	
Polyethylene oxide solution (ml)	—	5	—	
Ammonium chloride (g)	_	_	1	
Sodium carbonate (g)	1.75	1.75	1.75	
Sodium chloride (g)	0.25	0.25	0.25	
Sodium peroxide (g)	3	3	_	

Table A.1Composition of the compensating solutions for a volume
of 500 ml

A.4.2.38.5 Construction of calibration curve

Add from a burette the volumes of the silica dioxide standard solutions given in table A.2 into 100-ml polyethylene beakers each containing a magnetic stirrer bar.

Add 20 ml of the compensating solution by volumetric pipette and make up to 40 ml with water, of the volume as shown in table A.2, from a burette. While stirring with a magnetic stirrer, add 15 drops of hydrofluoric acid (1+3) (A.4.2.7). Stir for at least 1 min. Then add, from a volumetric pipette, 15 ml of the boric acid solution (A.4.2.29) with a volumetric pipette into the solution.

Add, from a volumetric pipette, 5 ml of the ammonium molybdate solution (**A.4.2.32**). Adjust the pH of this solution to 1.60 ± 0.05 by adding, drop by drop, sodium hydroxide solution (4 mol/L) (**A.4.2.18**) or hydrochloric acid (1+2) (**A.4.2.2**) using the pH meter calibrated with a buffer solution of similar pH [e.g. pH 1.40 (**A.4.2.37**)]. Transfer the solution to a 100-ml volumetric flask and rinse the beaker with hydrochloric acid (**A.4.2.5**) whose pH is 1.60 ± 0.05 . After 20 min, add, from a volumetric pipette, 5 ml of the citric acid solution (**A.4.2.30**), stir and leave to stand for 5 min. Then add, from a volumetric pipette, 2 ml of the reducing solution (**A.4.2.36**). Consider this as time 0.

Make up to the mark with dilute hydrochloric acid (**A.4.2.5**) whose pH is 1.60 ± 0.05 and mix. At time (0+30) min measure the absorbance with the absorption photometer (**A.5.8**) using a 10-mm absorption cell (**A.5.9**) against the blank solution prepared in the same way, using the wavelength of around 815 nm. Construct a curve giving the measured absorbance as a function of the corresponding silicon dioxide contents given in table A.2.

Determine the silicon dioxide content $(mgSiO_2/100 \text{ ml})$ from the calibration curve.

Serial No.	Blank test	1	2	3	4
SiO_2 standard solution (ml)	0	2	5	10	20
Water (ml)	20	18	15	10	0
SiO_2 content (mgSiO_2/100 ml)	0	0.04	0.10	0.20	0.40

Table A.2Amount of the silicon dioxide calibration solutions
and their silicon dioxide content

A.4.2.39 Calcium standard solution (approximately 0.01 mol/L)

Weigh, to ± 0.0005 g, $1.00 \text{ g} \pm 0.01 \text{ g}$ of calcium carbonate (**A.4.2.31**), and place it in a 400-ml beaker with approximately 100 ml of water. Cover the beaker with a watch glass and carefully introduce approximately 10 ml of hydrochloric acid (1+2) (**A.4.2.2**). Stir with a glass rod and ensure that dissolution is complete, bring to the boil in order to expel the dissolved carbon dioxide. Cool to room temperature, wash the solution into a 1 000-ml volumetric flask, and make up to the mark with water.

A.4.2.40 Ethylenediaminetetra-acetic acid disodium salt dihydrate (EDTA) solution (approximately 0.03 mol/L)

A.4.2.40.1 Preparation

Dissolve 11.17 g \pm 0.01 g of EDTA in water to make 1 000 ml. Store the solution in a polyethylene bottle.

A.4.2.40.2 Standardization

Place 50 ml of calcium standard solution (approximately 0.01 mol/L) (**A.4.2.39**) in a beaker suitable for the apparatus for photometric titration (**A.5.10**). Then dilute with water to a volume suitable for the operation of the apparatus.

Using the pH meter (A.5.16.1), adjust the pH of this solution to 12.5 ± 0.2 with either of the sodium hydroxide solutions (A.4.2.18 or A.4.2.19).

Perform the titration using one of the following two methods.

a) Photometric determination of the end-point (reference method)

Add about 0.1 g of murexide indicator (A.4.2.44) or of mixed calcein and methylthymol blue indicator (A.4.2.50). Place the beaker in the apparatus for photometric titration (A.5.10).

Set the apparatus at 620 nm when using murexide indicator (A.4.2.44) or at 520 nm when using the mixed indicator (A.4.2.50) and, while stirring continuously, titrate with EDTA solution (approximately 0.03 mol/L). In the vicinity of the indicator colour change, construct a curve giving the absorbance values as a function of the volume of EDTA solution (approximately 0.03 mol/L) added. The volume, V_3 , used is determined from the intersection of the line of greatest slope near the colour change and the line of almost constant absorbance after the colour change.

Calculate the factor f_D of the EDTA solution from formula (A.1):

- where, m_4 : mass of calcium carbonate required for preparation of calcium standard solution (A.4.2.39) (g)
 - V₃: volume of EDTA solution (approximately 0.03 mol/L) used for titration (ml)

b) Visual determination of the end-point (alternative method)

Add approximately 0.1 g of either calcon indicator (**A.4.2.46**) or the Patton and Reeders indicator (**A.4.2.51**). Stir the sample solution and titrate with EDTA solution (approximately 0.03 mol/L) until the colour changes from pink to blue, in the case of calcon indicator, and from purple to blue, in the case of the Patton and Reeders indicator. Take the point where one drop in excess does not further increase the intensity of the blue colour.

Calculate the factor f_D of the EDTA solution (approximately 0.03 mol/L) according to formula (A.2).

A.4.2.41 Copper complexonate solution

Take a 25-ml aliquot of the copper sulfate solution (**A.4.2.33**) into a 400-ml beaker using a volumetric pipette, and add correctly the EDTA solution (approximately 0.03 mol/L) (**A.4.2.40**) using a burette. Calculate the volume (V_5) of EDTA solution (approximately 0.03 mol/L) to be added as follows.

Take a 10-ml aliquot of the copper sulfate solution (**A.4.2.33**) into a 600-ml beaker using a volumetric pipette, make 200 ml with water, and add 10 ml of aqueous ammonia (**A.4.2.14**) and approximately 0.1 g of murexide indicator (**A.4.2.44**). Stir the sample solution and titrate with the EDTA solution (approximately 0.03 mol/L) (**A.4.2.40**). The end point shall be when the colour of the solution turns from pink to purple (V_4).

Calculate the volume V_5 of the EDTA solution (approximately 0.03 mol/L) that is necessary to add to 25 ml of copper sulfate solution to obtain copper complexonate solution from formula (A.2):

 $V_5 = 2.5 \times V_4$ (A.2)

where, V_4 : volume of EDTA solution (approximately 0.03 mol/L) used for titration (ml)

A.4.2.42.1 Preparation

Dissolve 11.40 g±0.01 g of EGTA in 400 ml of water and 30 ml of the sodium hydroxide solution (2 mol/L) (**A.4.2.19**) in a 600-ml beaker. Heat the mixture until the EGTA is completely dissolved. Allow to cool to room temperature. Using the pH meter (**A.5.16.1**), adjust the pH to 7.0 ± 0.5 , by adding, drop by drop, hydrochloric acid (1+2) (**A.4.2.2**). Transfer the solution quantitatively to a 1 000-ml volumetric flask and make up to the mark with water. Store the solution in a polyethylene bottle.

A.4.2.42.2 Standardization

Take a 50-ml aliquot of the calcium standard solution (approximately 0.01 mol/L) (A.4.2.39) into a beaker suitable for the apparatus for photometric titration (A.5.10) using a volumetric pipette. Then dilute with water to a volume suitable for the correct operation of the apparatus. Add 25 ml of the triethanolamine (1+4) solution (A.4.2.35).

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Using the pH meter (A.5.16.1), adjust the pH of this solution to 12.5 ± 0.2 with either of the sodium hydroxide solutions (A.4.2.18 or A.4.2.19).

Add about 0.1 g of murexide indicator (A.4.2.44) or of calcein indicator (A.4.2.45). Place the beaker in the apparatus for photometric titration (A.5.10), and titrate with the EGTA solution (approximately 0.03 mol/L) while stirring continuously.

The apparatus shall be set at 620 nm when using murexide indicator (**A.4.2.44**) or at 520 nm when using calcein indicator (**A.4.2.45**). In the vicinity of the indicator colour change, record the absorbance values and the correspondent volumes of EGTA solution (approximately 0.03 mol/L) added and construct a curve of absorbance versus volume of titrant. The volume V_6 used is determined from the intersection of the line of greatest slope near the colour change and the line of almost constant absorbance after the colour change.

Calculate the factor f_G of EGTA solution (approximately 0.03 mol/L) from formula (A.3):

where, m_5 : mass of calcium carbonate required for preparation of calcium standard solution (approximately 0.01 mol/L) (A.4.2.39) (g)

V₆: volume of EGTA solution (approximately 0.03 mol/L) used for titration (ml)

A.4.2.43.1 Preparation

Dissolve 3.64 g±0.01 g of DCTA in about 400 ml of water and 10 ml of sodium hydroxide solution (2 mol/L) (**A.4.2.19**) in a 600-ml beaker. Heat the mixture until the DCTA is completely dissolved. Allow to cool to room temperature. Using the pH meter (**A.5.16.1**), adjust the pH to 7.0 ± 0.5 by adding hydrochloric acid (1+2) (**A.4.2.2**), drop by drop. Transfer the solution quantitatively to a 1 000-ml volumetric flask and make up to the mark with water. Store this solution in a polyethylene bottle.

A.4.2.43.2 Standardization

Take a 50-ml aliquot of the calcium standard solution (approximately 0.01 mol/L) (A.4.2.39) into a beaker appropriate for the apparatus for photometric titration (A.5.10). Then dilute with water to a volume suitable for the correct operation of the apparatus.

Using the pH meter (A.5.16.1), adjust the pH of this solution to 10.5 ± 0.2 with aqueous ammonia (A.4.2.14).

Add about 0.1 g of murexide indicator (A.4.2.44) or of calcein indicator (A.4.2.45). Place the beaker in the apparatus (A.5.10) and, while continuously stirring the solution, titrate with the DCTA solution (approximately 0.01 mol/L).

The apparatus shall be set at 620 nm when using murexide indicator (A.4.2.44) or at 520 nm when using calcein indicator (A.4.2.45). In the vicinity of the colour change of the indicator, record the absorbance values and the correspondent volumes of DCTA solution (approximately 0.01 mol/L) added and construct a curve of absorbance versus volume of titrant. The volume, V_7 , used is determined by the intersection of the line of greatest slope near the colour change and the line of almost constant absorbance after the colour change.

Calculate the factor $f_{\rm C}$ of the DCTA solution from formula (A.4):

- where, m_6 : mass of calcium carbonate required for preparation of calcium standard solution (approximately 0.01 mol/L) (A.4.2.39) (g)
 - *V*₇: volume of DCTA solution (approximately 0.01 mol/L) used for titration (ml)

A.4.2.44 Murexide indicator Prepare by grinding 1.0 g \pm 0.1 g of murexide (ammonium purpurate, $C_8H_4N_5O_6NH_4$) with 100 g \pm 1 g of sodium chloride (A.4.2.23).

A.4.2.45 Calcein indicator Prepare by grinding 1.0 g \pm 0.1 g of calcein {bis[(bis (carboxymethyl)-amino-methyl)]-2',7'-fluorescein, $C_{30}H_{26}N_2O_{13}$ } with 100 g \pm 1 g of potassium nitrate (KNO₃).

A.4.2.47 Sulfosalicylic acid indicator (5-sulfosalicylic acid dihydrate)

A.4.2.48 PAN indicator Prepare by dissolving 0.10 g \pm 0.01 g of PAN [1-(2-pyridylazo)-2-naphthol, C₁₅H₁₁N₃O] in 100 ml \pm 1 ml of ethanol (C₂H₅OH, ρ =0.79 g/cm³).

A.4.2.49 Methylthymol blue indicator Prepare by grinding 1.0 g \pm 0.1 g of methylthymol blue {sodium salt of 3',3"-bis-[bis(carboxy-methyl)-aminomethyl]-thymolsulfophthalein, $C_{37}H_{41}N_2O_{13}SNa_3$ } with 100 g \pm 1 g of potassium nitrate (KNO₃).

A.4.2.50 Mixed calcein and methylthymol blue indicator Prepare by grinding $0.20 \text{ g} \pm 0.02 \text{ g}$ of calcein (**A.4.2.45**) and $0.10 \text{ g} \pm 0.01 \text{ g}$ of methylthymol blue (**A.4.2.49**) with $100 \text{ g} \pm 1 \text{ g}$ of potassium nitrate (KNO₃).

A.4.2.51 Patton and Reeders reagent Prepare by mixing 1.0 g \pm 0.1 g of [2-hydroxy-1-(2-hydroxy-4-sulfo-1-napthylazo)-3-napthoic acid, $C_{21}H_{14}N_2O_7S$] with 100 g \pm 1 g of anhydrous sodium sulfate (Na₂SO₄).

A.4.2.52 Mixed indicator Prepare by mixing 0.10 g \pm 0.01 g of o-cresophthalein comlexone [o-cresolphthaleindi-(methyliminodi-acetic acid), $C_{32}H_{32}N_2O_{12}$], 0.020 g \pm 0.001 g of methyl red (o-carboxybenzene-azodimethyl-aniline, $C_{15}H_{14}N_3NaO_2$), 0.030 g \pm 0.001 g of naphtol green B ($C_{30}H_{15}FeN_3Na_3O_{15}S_3$) with 10.0 g \pm 0.1 g of sodium chloride (A.4.2.23).

A.5 Apparatus

A.5.1 Balance(s), capable of weighing to an accuracy of ± 0.0005 g.

A.5.2 Crucibles

A.5.2.1 Crucibles, porcelain or platinum, 20 ml to 25 ml capacity.

NOTE : The methods in this Annex specify where it is necessary to use platinum crucibles. Unless platinum is specified, porcelain crucibles can be used.

A.5.2.2 Lids, suitable lids to be fitted to crucibles (A.5.2.1), where required.

A.5.3 Ceramic support(s), fire-proof, or preventing overheating of the crucible. It shall be in thermal equilibrium with the furnace at the moment the crucible is introduced.

A.5.4 Evaporating dish, porcelain, with approximately 200 ml capacity.

A.5.5 Electric furnace(s), capable of being set at the following temperatures: 500 °C ± 10 °C, 950 °C ± 25 °C and 1 175 °C ± 25 °C.

A.5.6 **Desiccator**(s), containing anhydrous magnesium perchlorate $Mg(ClO_4)_2$ or silica gel.

NOTE : Where self-indicating silica gel is used, a non-toxic indicator is recommended.

A.5.7 Oven(s), capable of being set at the following temperatures: 110 °C±5 °C, 120 °C ±5 °C, 150 °C±5 °C, 200 °C±10 °C and 250 °C±10 °C.

A.5.8 Absorption photometer(s), for measuring the absorbance of a solution in the vicinity of 525 nm and 815 nm.

A.5.9 Cells, for the absorption photometer.

A.5.10 Apparatus for photometric titration, capable of measuring the absorbance at 520 nm and 620 nm of a solution contained in a titration beaker, while stirring with a magnetic stirrer.

A.5.11 Stirrer, e.g. magnetic stirrer, with inert, e.g., PTFE, covered bar.

A.5.12 Evaporation apparatus, controlled at 105 °C \pm 5 °C, e.g. water bath or hot plate.

A.5.13 Sand bath or hot plate, controlled at approximately 400 °C.

A.5.14 Filter paper, ashless.

NOTE : Filter papers with a mean pore diameter of around 2 μ m are termed fine, those with a mean pore diameter around 7 μ m are termed medium and those with a mean pore diameter of around 20 μ m are termed coarse.

A.5.15 Volumetric glassware, of analytical accuracy, e.g. Class A as defined in ISO 385 and ISO 835.

A.5.16 pH measuring equipment

A.5.16.1 pH meter, capable of measuring to an accuracy of ± 0.05 .

A.5.16.2 pH indicator paper(s), capable of measuring pH in the 0 to 14 range.

A.6 Preparation of test sample of cement

Before chemical analysis, treat the laboratory sample as follows to obtain a homogeneous test sample:

Take approximately 100 g of the laboratory sample by means of a sample divider or by quartering. Sieve this portion on a 150 μ m or 125 μ m sieve until the residue remains constant. Remove metallic iron from the materials retained on the sieve by means of a magnet. Then grind the iron free fraction of the retained material so that it completely passes the 150 μ m or 125 μ m sieve. Transfer the sample to a clean, dry container with an airtight closure and shake vigorously to mix it thoroughly.

Carry out all operations as quickly as possible to ensure that the test sample is exposed to ambient air only for the minimum time.

NOTE : Where the analysis is one of a series subject to statistical control and the level of the metallic iron content has been shown to be insignificant in relation to the chemical properties being determined, then it is not necessary to remove metallic iron.

A.7 to A.12 (Deleted since identical to the corresponding clauses in the text and Annex B).

A.13 Determination of major elements

A.13.1 Principle

The analysis is carried out after the cement is completely dissolved. The decomposition with hydrochloric acid and ammonium chloride (alternative method) may be used for cement with an insoluble residue [as determined in accordance with hydrochloric acid-sodium carbonate method (clause 6)] not exceeding 1.5 %.

The cement is decomposed by sintering (melting) with sodium peroxide or by treatment with hydrochloric acid in the presence of ammonium chloride. In the first case, after dissolution of the sintered solid (melt) in hydrochloric acid, the major part of the silicon dioxide is precipitated either by double evaporation or by hydrochloric acid with coagulation by polyethylene oxide; in the second case, the major part of the silicon dioxide is separated by the treatment. The impure silicon dioxide precipitated is treated with hydrofluoric acid and sulfuric acid to volatilize silicon dioxide; the residue, treated with a mixture of sodium carbonate and sodium chloride, is dissolved in hydrochloric acid and added to the silicon dioxide filtrate.

In the case of the treatment with hydrochloric acid in the presence of ammonium chloride, if the residue obtained after volatilization of impure silicon dioxide by means of hydrofluoric acid and sulfuric acid is greater than 0.5 %, the method is not applicable. In this case, it is necessary to decompose the cement by sodium peroxide.

In the final solution, the soluble silicon dioxide is determined by photometric determination, and iron (III) oxide, aluminium oxide, calcium oxide and magnesium oxide are determined by complexometric methods.

The schematic diagram of the chemical analysis is shown in figure A.1.

The relative amounts of impure, pure and soluble silicon dioxide may vary depending on the procedure used, but the same result for the total silicon dioxide is obtained whichever path in figure A.1 is chosen.