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Magnesites and dolomites — Chemical analysis

Produits de magnésie et de dolomie — Analyse chimique

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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.

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.

International Standard ISO 10058 was prepared by Technical Committee ISO/TC 33, *Refractories*, Sub-Committee SC 2, *Methods of testing*.

Annex A forms an integral part of this International Standard.

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Magnesites and dolomites — Chemical analysis

1 Scope

This International Standard specifies methods for the determination of silica, alumina, titania, iron oxide and oxides of manganese, chromium, calcium, magnesium, sodium, potassium and lithium. It also specifies methods for determining the loss on ignition of magnesite and dolomite, and of refractories based on these raw materials.

Annex A describes a method for the determination of the boron content of magnesites only.

NOTE 1 Physical methods are used increasingly for chemical analysis. At present, it is not possible to describe a standardized test method, because the type of apparatus used is important.

2 Dissolution and determination of silica

2.1 Principle

Decompose the sample with hydrochloric acid and separate the silica by coagulation with a polyethylene oxide solution. Filter, wash the residue, heat and weigh it, and submit it to a treatment with hydrofluoric and sulfuric acids. After this treatment, weigh the remaining residue again and fuse it in sodium carbonate and boric acid. It is then dissolved in the filtrate from the silica, and the solution is diluted to a standard volume to obtain the stock solution (A) of the sample.

In an aliquot, the small quantity of silica not separated by coagulation is subsequently determined by a spectrophotometric method based on the formation of molybdenum blue, using alternatively iron(II) sulfate or tin(II) chloride as a reducing agent. The absorbance maximum of the reduced silicomolybdate complex lies at a wavelength of 810 nm.

2.2 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

2.2.1 Sodium carbonate, anhydrous.

2.2.2 Boric acid, powdered.

2.2.3 Polyethylene oxide solution, 2,5 g/l.

Add 0,5 g of polyethylene oxide to 200 ml of water while stirring slowly, preferably with a mechanical stirrer, until dissolved. Discard after 2 weeks.

2.2.4 Accelerator granules, ashless, of mass about 1 g.

2.2.5 Hydrochloric acid, concentrated, $\rho = 1,19$ g/ml.

2.2.6 Hydrochloric acid, diluted 1 + 19.

Add 1 volume of hydrochloric acid (2.2.5) to 19 volumes of water.

2.2.7 Sulfuric acid, concentrated, $\rho = 1,84$ g/ml.

2.2.8 Hydrofluoric acid, 40 % (m/m).

2.3 Apparatus

Usual laboratory apparatus and the following.

2.3.1 Sand bath or hot plate.

2.3.2 Muffle furnace, capable of being controlled at 1 180 °C to 1 200 °C.

2.3.3 Platinum crucible.

2.4 Procedure

Weigh into a 250 ml beaker, 5,000 g¹⁾ of finely ground analytical sample dried at 110 °C. Add 25 ml of water and 40 ml of concentrated hydrochloric acid (2.2.5) and cover with a watch-glass. Transfer to a sand bath or a hot-plate (2.3.1) and boil for 30 min. Allow the beaker and contents to cool, rinse the watch-glass with water, add the accelerator granules (2.2.4) and stir to break up the pulp. Then add, stirring all the time, 5 ml of polyethylene oxide solution (2.2.3) and allow to stand for 5 min. Filter the solution through a closed-pore paper filter suitable for separating very fine precipitates. Transfer the residue (containing the precipitate of silica) quantitatively with hot diluted hydrochloric acid (2.2.6) to the filter paper. Wash the precipitate six times with hot diluted hydrochloric acid (2.2.6) and then with hot water until free from chlorides (up to a volume of about 400 ml). Store the filtrate. Transfer the filter paper and precipitate to a heated and weighed platinum crucible (2.2.3). Heat at a low temperature until the precipitate is free from carbonaceous matter, then heat it in the muffle furnace (2.3.2) controlled at 1 180 °C to 1 200 °C to constant mass (m_1), a time of 15 min normally being sufficient.

Moisten the residue in the cold crucible with water, add 5 drops of concentrated sulfuric acid (2.2.7) and 10 ml of hydrofluoric acid (2.2.8). Evaporate to dryness in the sand bath or on the hot-plate in a fume cupboard.

Heat the crucible and the residue, cautiously at first over a Bunsen burner, and finally for 5 min between 1 180 °C and 1 200 °C in the muffle furnace. Allow to cool in a desiccator and weigh (mass m_2). If the residue weighs more than 30 mg, repeat the treatment with hydrofluoric and sulfuric acids to ensure that all the silica is removed. The difference between the two masses ($m_1 - m_2$) represents the "gravimetric" silica.

Fuse the residue from the hydrofluoric and sulfuric acid treatment with 2 g of the anhydrous powdered sodium carbonate (2.2.1) and 0,7 g of powdered boric acid (2.2.2). Allow the melt to cool and dissolve in the filtrate from the main silica. Cool, transfer the solution to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

This solution will become stock solution A of the sample for the spectrophotometric determination of the residual silica in accordance with 2.5.1 or 2.5.2 and for the determination of alumina (clause 3), iron(III) oxide (clause 4), titanium(IV) oxide (clause 5), manganese(IV) oxide (clause 6), chromium(III) oxide (clause 7), calcium oxide (clause 8) and magnesium oxide (clause 9).

2.5 Spectrophotometric determination of residual silica in filtrate

2.5.1 Reduction to molybdenum blue complex with iron(II) sulfate

2.5.1.1 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

2.5.1.1.1 Ammonium hydroxide solution,

$\rho = 0,9\text{g/ml}$.

Although the method allows for the presence of silica in the ammonium hydroxide solution, the reagent should contain as little silica as possible. It should be noted that ammonium hydroxide solution stored in glass bottles will dissolve silica from the glass.

2.5.1.1.2 Ammonium iron(III) sulfate solution.

Dissolve 100 g of ammonium iron(III) sulfate dodecahydrate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ in water, add 300 ml of concentrated hydrochloric acid (2.2.5) and dilute to 1 litre with water.

2.5.1.1.3 Ammonium molybdate solution.

Dissolve 80 g of hexammonium heptamolybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in water, filter if necessary, dilute to 1 litre and mix. Store in a polyethylene bottle. Renew the solution after 4 weeks, or earlier if any appreciable deposit is observed.

2.5.1.1.4 Oxalic acid solution.

Dissolve 100 g of oxalic acid dihydrate ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) with water and dilute to 1 litre.

2.5.1.1.5 Iron(II) sulfate solution.

Dissolve 10 g of iron(II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in water, add 4 drops of diluted sulfuric acid (1 + 1) and dilute to 100 ml with water. Prepare this solution freshly before use.

2.5.1.1.6 Silica stock solution, containing 0,5 g of SiO_2 per litre.

Ignite a sample of high purity silica to 1 000 °C and cool. Then weigh 0,250 g of this sample in a platinum crucible with 5 g of anhydrous sodium carbonate. Dissolve the melt in a polyethylene vessel in 300 ml of water while adding 20 g of sodium hydroxide.

1) In the case of natural iron and silica-rich magnesite, the sample mass may optionally be reduced to 2,000 g. The aliquots to be taken for the determination are to be changed accordingly.

Cool the solution to 20 °C, transfer to a 500 ml one-mark volumetric flask and make up to the mark with water. Stir thoroughly and transfer to a polyethylene flask for storage. This solution will remain stable for 6 months.

1 ml of this standard solution contains 0,5 mg of SiO₂.

2.5.1.1.7 Silica standard solution, containing 0,02 g of SiO₂ per litre.

Dilute 20 ml of the silica stock solution (2.5.1.1.6) to 500 ml with water in a one-mark volumetric flask. Prepare this solution daily if required.

1 ml of this standard solution contains 0,02 mg of SiO₂.

2.5.1.1.8 Phenolphthalein solution.

Dissolve 100 g of phenolphthalein in ethanol and dilute to 100 ml.

2.5.1.2 Apparatus

Usual laboratory apparatus and a spectrophotometer, fitted with cells of an appropriate size.

2.5.1.3 Procedure

2.5.1.3.1 Pipette two 5 ml aliquots of the stock solution A, prepared as in 2.4, into two 100 ml one-mark volumetric flasks. Add one drop of phenolphthalein solution (2.5.1.1.8) to each flask, and while continually shaking, add ammonium hydroxide solution (2.5.1.1.1) one drop at a time. As soon as the alkaline reaction produces a red colour, immediately add 5 ml of ammonium iron(III) sulfate solution (2.5.1.1.2). To the first of the two solutions, add 5 ml of ammonium molybdate solution (2.5.1.1.3) and leave to stand for 20 min.

NOTE 2 Cloudiness may occur but it does not cause any interference and disappears after the addition of oxalic acid.

Add 20 ml of oxalic acid solution (2.5.1.1.4) and shake the solution until it is clear. Add 1 ml of iron(II) sulfate solution and make up to the mark with water. Take the second 5 ml aliquot part of the stock solution A (see 2.4) which will serve as a reference, add 20 ml of oxalic acid solution to it and leave it also to stand for 20 min. Add 5 ml of ammonium molybdate solution, then 1 ml of iron(II) sulfate solution and make up to the mark with water. Carry out a blank determination on a solution containing all the reagents, but omitting the stock solution (see 2.4).

2.5.1.3.2 Measure, in cells of an appropriate size, the absorbance of the solution brought to 20 °C ± 2 °C, using the optimum wavelength of 810 nm, and compare it with the reference solution

(2.5.1.3.1). Subtract the absorbance of the blank solution from that of the sample solution.

2.5.1.3.3 The blank solution is treated and measured in the same manner as the sample. Subtract the absorbance of the blank solution from that of the sample solution.

2.5.1.4 Plotting the calibration graph.

Pipette 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml volumes of the silica standard solution (2.5.1.1.7) into six 100 ml one-mark volumetric flasks.

Add one drop of the phenolphthalein solution (2.5.1.1.8) to the contents of each flask and while stirring constantly, mix with hydrochloric acid (1 + 4) until the red colour disappears. Then add the ammonium hydroxide solution (2.5.1.1.1) drop by drop. As soon as the alkaline reaction produces a red colour, immediately mix with 5 ml of the ammonium iron(III) sulfate solution (2.5.1.1.2) and 5 ml of the ammonium molybdate solution (2.5.1.1.3), and allow to stand for 20 min. Add 20 ml of the oxalic acid solution (2.5.1.1.4) and shake the solution until it becomes clear. Add 1 ml of the iron(II) sulfate solution (2.5.1.1.5) and make up to the mark with water.

Measure the absorbance of each calibration solution maintained at 20 °C ± 2 °C using the spectrophotometer set at an optimum wavelength of 810 nm against the zero member of the set of calibration solutions as a reference.

Use the values thus obtained to plot a calibration graph. On the basis of this graph, determine the mass of silica contained in 5 ml of the stock solution A.

2.5.1.5 Expression of results

The silica content, $w(\text{SiO}_2)$, expressed as a percentage by mass, is given by the following formula:

$$w(\text{SiO}_2) = \frac{(m_1 - m_2) + (m_3 - m_4)}{m_0} \times 100$$

where

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in grams, of the crucible and its contents before treatment with hydrofluoric and sulfuric acids;
- m_2 is the mass, in grams, of the crucible with the residue after treatment with hydrofluoric and sulfuric acids;
- m_3 is the mass, in grams, of silica contained in 5 ml of the stock solution A;

m_4 is the mass, in grams, of silica found in the blank test solution.

2.5.2 Reduction to molybdenum blue complex by tin(II) chloride

2.5.2.1 Reagents, (additional to 2.5.1)

2.5.2.1.1 2,4-Dinitrophenol solution.

Dissolve 0,1 g of 2,4-dinitrophenol in hot water, dilute to 100 ml, cool and filter.

2.5.2.1.2 Tin(II) chloride solution.

Dissolve 1 g of tin(II) chloride in 1,5 ml of hydrochloric acid (2.2.5), cool and dilute to 100 ml. Prepare a fresh solution when required.

2.5.2.2 Procedure

Pipette 5 ml of stock solution A, obtained as in 2.4 into a 100 ml volumetric flask (A). Add 15 ml of water and two drops of the dinitrophenol solution (2.5.2.1.1), then add dilute ammonia solution (1 + 1) drop by drop until the indicator changes to yellow. Note the number of drops required. Then add 5 ml of dilute hydrochloric acid (1 + 4).

Take a second 100 ml volumetric flask (B), pipette 20 ml of water into it and add the same amount of ammonia solution (1 + 1) as that used for neutralizing in flask A. Add two drops of the dinitrophenol solution (2.5.2.1.1) and then add dilute hydrochloric acid (1 + 4) until the indicator changes to colourless. Then add 5 ml of dilute hydrochloric acid (1 + 4) in excess.

Add 6 ml of ammonium molybdate solution (2.5.1.1.3) to each volumetric flask (A and B) and allow to stand for 5 min to 10 min at 20 °C to 30 °C. Then add 45 ml of dilute hydrochloric acid (1 + 1) and allow to stand for a further 10 min. After adding 10 ml of tin(II) chloride solution (2.5.2.1.2), make up to 100 ml and mix thoroughly.

Measure, in cells of convenient size, the absorbance of the solution in flask A against that of the solution in flask B using suitable apparatus and an optimum wavelength of 810 nm. The colour will remain stable for 5 min to 30 min after adding tin(II) chloride solution.

2.5.2.3 Plotting the calibration graph

Pipette the following volumes into six 100 ml volumetric flasks.

0	2	4	6	8	10	ml silica standard solution (2.5.1.1.7)
10	8	6	4	2	0	ml distilled water

Add to each flask, while shaking, 5 ml of dilute hydrochloric acid (1 + 4) and 6 ml of ammonium molybdate solution (2.5.1.1.3) and allow to stand for 5 min to 10 min at 20 °C to 30 °C. Then add 45 ml of dilute hydrochloric acid (1 + 1) while shaking and leave to stand for a further 10 min. After adding 10 ml of tin(II) chloride solution, fill up to 100 ml and mix thoroughly.

Measure the absorbances of the solutions with additions of silica against the zero solution. Use suitable equipment and the wavelength of 810 nm (optimum). The colour will remain stable for 5 min to 10 min after adding tin(II) chloride solution.

2.5.2.4 Calculation

Calculate "gravimetric" silica from using the difference between the masses m_1 and m_2 obtained according to 2.4. Calculate the total silica content by adding the silica remaining in the stock solution A to the "gravimetric" silica.

3 Determination of alumina

3.1 Principle

Dilute an aliquot of stock solution A, prepared as in 2.4, and take an aliquot from this diluted stock solution. Add 8-hydroxyquinoline solution and buffer with ammonium acetate solution. Extract the aluminium oxinate formed by means of chloroform and measure the extinction of the yellow extract solution. The absorbance maximum of aluminium oxinate in chloroform lies at a wavelength of 390 nm. Under these conditions, the Lambert-Beer law is obeyed up to a maximum concentration of 0,2 mg Al_2O_3 in the measured solution (extract).

Iron, forming a black oxinate also soluble in chloroform, is converted to the iron(II) phenanthroline complex which does not react with oxine.

The method is suitable for determination of alumina contents up to 30 %. Other elements contained in magnesite and dolomite do not interfere with the determination if they do not exceed the usual concentrations. However, when more than 0,2 % titanium oxide is present, alumina values which are too high will be found due to extraction of titanium(IV) oxide, but this is eliminated by complexing titanium(IV) oxide by addition of hydrogen peroxide.

3.2 Reagents

3.2.1 8-Hydroxyquinoline solution.

Dissolve 5 g of 8-hydroxyquinoline ("oxine") in 50 ml of hydrochloric acid (1 volume of hydrochloric acid (2.2.5) to 1 volume of water), while heating. After cooling, dilute with water to one litre.

3.2.2 Thioglycolic acid.

Mix equal volumes of thioglycolic acid (80 %) and water. Store in a dropper.

3.2.3 1,10-Phenanthroline solution.

Dissolve 1 g of 1,10-phenanthroline chloride in water and dilute to 250 ml.

NOTE 3 The solution will keep for about 1 week.

3.2.4 Ammonium acetate solution.

Dissolve 200 g of ammonium acetate in water and dilute to 1 litre.

3.2.5 Chloroform.

3.2.6 Ethylene glycol mono-*n*-butylether.

3.2.7 Sodium hydroxide solution.

Dissolve 100 g of sodium hydroxide in water and dilute to 1 litre.

3.2.8 Alumina stock solution (1 mg Al₂O₃/ml).

Dissolve 0,529 g of pure metallic aluminium in a covered polyethylene beaker in 50 ml of sodium hydroxide solution. Heat gently if necessary. Then add 100 ml of hydrochloric acid (2.2.5) and heat until the solution becomes completely clear. Cool to 20 °C and dilute with water in a volumetric flask to 1 000 ml.

3.2.9 Aluminium standard solution

(0,01 mg Al₂O₃/ml).

Dilute 10 ml of the alumina stock solution (3.2.8), after addition of 25 ml of hydrochloric acid (2.2.5), with water to 1 000 ml in a volumetric flask. Prepare a fresh solution daily if required.

3.3 Procedure

Pipette 20 ml of stock solution A, prepared as in 2.4, into a 250 ml volumetric flask and dilute to volume with water. Pipette an aliquot, the amount of which depends on the alumina content of the diluted stock solution, into a separating funnel of volume 100 ml.

For an alumina content up to and including

1 %: use 20 ml of diluted stock solution, prepared from the stock solution A;

2 %: use 10 ml of diluted stock solution, prepared from the stock solution A;

5 %: use 5 ml of diluted stock solution, prepared from the stock solution A.

If the alumina content exceeds 5 %, dilute the stock solution so that the concentration of the aliquot to be taken (5 ml or 10 ml) does not exceed 0,2 mg Al₂O₃.

Dilute the aliquot in the separating funnel, if necessary, to 20 ml with water and add 5 drops of thioglycolic acid (3.2.2) and 10 ml of 1,10-phenanthroline (3.2.3) solution. Then add in the following order: 10 ml of 8-hydroxyquinoline solution (3.2.1), 1 ml of ethylene glycol mono-*n*-butylether (3.2.6) and 10 ml of ammonium acetate solution (3.2.4). Shake vigorously for 2 min, after addition of 25 ml of chloroform (from a pipette with automatic zero adjustment or a burette).

After the phase separation, drain off some of the organic phase to rinse the stop-cock and outlet. Then close the stopcock and place a wad loosely in the outlet tube, to retain drops of water. Rinse again with some of the extract. Without interruption, run out the chloroform extract into a dry 1 cm cell and cover immediately with a suitable lid. Measure the absorbance in a spectrophotometer at a wavelength of 390 nm, using water as a reference solution.

Make a blank measurement using the reagents without the stock solution, and subtract this measurement from the result.

3.4 Plotting the calibration graph

Pipette 0 ml, 5 ml, 10 ml, 15 ml and 20 ml volumes of aluminium standard solution (3.2.9) into five 100 ml volumetric flasks, and proceed as in 3.3. Use these results to plot a calibration graph, making the necessary corrections for blank solutions (see 3.3).

3.5 Calculation

Calculate the alumina content, $w(\text{Al}_2\text{O}_3)$, expressed as a percentage by mass, using the calibration graph, as follows:

$$w(\text{Al}_2\text{O}_3) = \frac{m_1 V}{m_2 V_1} \times 100$$

where

m_1 is the amount of alumina in the sample solution, in milligrams, corrected for the blank value taken from the calibration graph;

- V is the volume, in millilitres, of diluted stock solution (see 3.3);
- m_2 is the mass, in milligrams, of sample contained in the aliquot of stock solution A, as used for preparation of the diluted stock solution;
- V_1 is the aliquot, in millilitres, taken from the diluted stock solution.

4 Determination of total iron calculated as iron(III) oxide

4.1 Principle

Prepare a second diluted stock solution (B) from the stock solution A prepared as in 2.4. This also serves for the determination of calcium and magnesium. By adding hydroxylammonium chloride to an aliquot of this diluted stock solution, reduce the iron to its divalent form. On adding 1,10-phenanthroline and buffering with ammonium acetate, an orange-coloured ferrous phenanthroline complex will form, the intensity of which is determined photometrically. The absorbance maximum of this complex lies at a wavelength of 510 nm. By an appropriate dilution of the measuring solution iron(III) contents up to about 15 % may be determined.

4.2 Reagents

4.2.1 Ammonium acetate solution.

See 3.2.4.

4.2.2 Hydroxylammonium chloride solution.

Dissolve 5 g of hydroxylammonium chloride in 250 ml of water.

4.2.3 1,10-phenanthroline solution.

See 3.2.3.

4.2.4 Iron(III) oxide stock solution

(1,2 mg Fe₂O₃/ml).

Weigh into a beaker 1,2 g ± 0,001 g of iron(III) oxide, which has been dried at 110 °C ± 2 °C to constant mass, and dissolve, while heating gently with 25 ml of hydrochloric acid (2.2.5). Cool to 20 °C and dilute to 1 000 ml with water in a volumetric flask.

4.2.5 Iron(III) oxide standard solution

(0,06 mg Fe₂O₃/ml).

Dilute 25 ml of the stock solution (4.2.4) to 500 ml with water in a volumetric flask.

4.3 Procedure

Dilute 50 ml of stock solution A, prepared as in 2.4, to 250 ml with water in a one-mark volumetric flask. Transfer 5 ml of this diluted stock solution (B) into a 100 ml one-mark volumetric flask. Then add in the following order: 10 ml of the hydroxylammonium chloride solution (4.2.2), 5 ml of phenanthroline (4.2.3) and 10 ml of ammonium acetate solution (4.2.1). Allow to stand for 15 min, make up to the mark with water and mix by shaking. Measure the absorbance of the solution at 20 °C ± 2 °C, at a wavelength of 510 nm, using water as a reference solution.

Make a blank measurement using the reagents without the stock solution, and subtract this measurement from the result.

4.4 Plotting the calibration graph

Pipette 0 ml, 1 ml, 2 ml, 4 ml, 5 ml and 10 ml volumes of iron(III) oxide standard solution (4.2.5) into six 100 ml volumetric flasks and proceed as in 4.3. Use these results to plot a calibration graph, making the necessary corrections for blank solutions (see 4.3).

4.5 Calculation

Calculate the iron(III) oxide content, $w(\text{Fe}_2\text{O}_3)$, expressed as a percentage by mass, using the calibration graph, as follows:

$$w(\text{Fe}_2\text{O}_3) = \frac{m_1 V}{m_2 V_1} \times 100$$

where

- m_1 is the mass of iron(III) oxide in the sample solution, in milligrams, corrected for the blank value taken from the calibration graph;
- V is the volume, in millilitres, of diluted stock solution (see 4.3);
- m_2 is the mass, in milligrams, of sample contained in the aliquot of stock solution A, as used for preparation of the diluted stock solution;
- V_1 is the aliquot, in millilitres, taken from the diluted stock solution.

5 Determination of titanium(IV) oxide

5.1 By formation of a complex with hydrogen peroxide

5.1.1 Principle

Mix an aliquot of stock solution A, prepared as in 2.4, with sulfuric acid and hydrogen peroxide solution, and measure the yellow colour photometrically.

Titanium(IV) oxide contents of up to 0,5 % can be measured. However, vanadium interferes because of the formation of a similar yellow coloured complex, and if more than 0,1 % vanadium pentoxide is present, the method given in 5.2 is used.

Interference may also be caused by deposits of zirconium phosphate, which should be removed by filtering or centrifuge.

5.1.2 Reagents

5.1.2.1 Hydrogen peroxide solution, 6 % (20 volumes).

5.1.2.2 Phosphoric acid solution, diluted 2 + 3.

Add 400 ml of phosphoric acid (1,7 g/ml) to 600 ml of water, then mix and allow to cool.

5.1.2.3 Sulfuric acid solution, diluted 1 + 4.

Add cautiously while stirring 100 ml of sulfuric acid (2.2.7) to 400 ml of water and allow to cool.

5.1.2.4 Titanium(IV) oxide stock solution, (1 mg TiO₂/ml)

Fuse 1 g of pure titanium(IV) oxide, dried to constant mass by ignition, with 10 g of potassium bisulphate. Allow to cool, and to avoid hydrolysis, dissolve the melt at a temperature below 50 °C, in 200 ml of water to which 20 ml of sulfuric acid (2.2.7) has been added. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.1.2.5 Titanium(IV) oxide standard solution, (0,04 mg TiO₂/ml).

Dilute 20 ml of titanium(IV) oxide stock solution (5.1.2.4) with water in a 500 ml volumetric flask, and mix. This solution should be prepared freshly when required.

5.1.3 Procedure

Pipette 40 ml of stock solution A, prepared as in 2.4, into each of two 100 ml volumetric flasks and mix with 5 ml of sulfuric acid (5.1.2.3) and 10 ml of phosphoric acid (5.1.2.2). Add 10 ml of hydrogen

peroxide solution to one of the flasks and allow to stand for 10 min, then fill up both flasks to 100 ml with water and stir vigorously.

Measure the absorbance of the solution at 20 °C ± 2 °C against the solution without the addition of hydrogen peroxide using a wavelength of 400 nm.

NOTE 4 The solution should be filtered or centrifuged to remove zirconium phosphate.

5.1.4 Plotting the calibration graph

Pipette 0 ml, 10 ml, 20 ml, 40 ml and 50 ml volumes of titanium(IV) oxide standard solution (5.1.2.5) into two series of five 100 ml volumetric flasks.

Add 5 ml of sulfuric acid (5.1.2.3) and 10 ml of phosphoric acid (5.1.2.2) to all the flasks. Then add to one of each pair of volumetric flasks, 10 ml of hydrogen peroxide solution (5.1.2.1) and allow to stand for 10 min, then fill up both flasks to 100 ml with water and stir vigorously. Measure the absorbance of the solution at 20 °C ± 2 °C, against the solution without the addition of hydrogen peroxide, using a wavelength of 400 nm (see also 5.1.3).

5.1.5 Calculation

Calculate the titanium(IV) oxide content, $w(\text{TiO}_2)$, expressed as a percentage by mass, using the calibration graph (5.1.4) as follows:

$$w(\text{TiO}_2) = \frac{m_1 - m_2}{m_0} \times 1,25$$

where

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in milligrams, of titanium(IV) oxide in the sample solution;
- m_2 is the mass, in milligrams, of titanium(IV) oxide in the blank solution.

5.2 By formation of a complex with chromotropic acid

5.2.1 Principle

After decomposition of the sample by hydrochloric acid and possible separation of silica, formation of an orange-red complex of the titanium(IV) oxide with chromotropic acid in a chloracetic buffer solution (pH about 2.9).

5.2.2 Reagents

5.2.2.1 Hydrochloric acid

See 2.2.5.

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