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Extenders for paints — Specifications and methods of test —

Part 20:
Fumed silica

Matières de charge pour peintures — Spécifications et méthodes d'essai —

Partie 20: Silice pyrogénée

Reference number
ISO 3262-20:2000(E)

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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 3.

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 part of ISO 3262 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3262-20 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders*.

Together with the other parts (see below), this part of ISO 3262 cancels and replaces ISO 3262:1975, which has been technically revised. Part 1 comprises the definition of the term extender and a number of test methods that are applicable to most extenders, whilst part 2 and the following parts specify requirements and, where appropriate, particular test methods for individual extenders.

ISO 3262 consists of the following parts, under the general title *Extenders for paints — Specifications and methods of test*:

- *Part 1: Introduction and general test methods*
- *Part 2: Barytes (natural barium sulfate)*
- *Part 3: Blanc fixe*
- *Part 4: Whiting*
- *Part 5: Natural crystalline calcium carbonate*
- *Part 6: Precipitated calcium carbonate*
- *Part 7: Dolomite*
- *Part 8: Natural clay*
- *Part 9: Calcined clay*
- *Part 10: Natural talc/chlorite in lamellar form*
- *Part 11: Natural talc, in lamellar form, containing carbonates*
- *Part 12: Muscovite-type mica*
- *Part 13: Natural quartz (ground)*

- *Part 14: Cristobalite*
- *Part 15: Vitreous silica*
- *Part 16: Aluminium hydroxides*
- *Part 17: Precipitated calcium silicate*
- *Part 18: Precipitated sodium aluminium silicate*
- *Part 19: Precipitated silica*
- *Part 20: Fumed silica*
- *Part 21: Silica sand (unground natural quartz)*
- *Part 22: Flux-calcined kieselguhr*

INTERNATIONAL STANDARD **ISO 3262-20:2000(E)**

Extenders for paints — Specifications and methods of test —

Part 20:
Fumed silica

1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for fumed silica.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 3262. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 3262 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 787-11:1981, *General methods of test for pigments and extenders — Part 11: Determination of tamped volume and apparent density after tamping.*

ISO 787-18:1983, *General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure.*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3262-1:1997, *Extenders for paints — Specifications and methods of test — Part 1: Introduction and general test methods.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3819:1985, *Laboratory glassware — Beakers.*

ISO 15528:—1), *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

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1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

3 Term and definition

For the purposes of this part of ISO 3262, the following term and definition apply:

3.1
fumed silica
amorphous silica produced from silicon halides by high-temperature flame hydrolysis

4 Requirements and test methods

For fumed silica complying with this part of ISO 3262, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2.

Table 1 — Essential requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Silica content, min.	% (*m*/*m*)	99,8		See clause 6
Oxide content				See clause 7
— Al_2O_3, max.		0,05		
— TiO_2, max.		0,03		
— Fe_2O_3, max.		0,003		
Carbon content[a]		max. 0,2	min. 0,3	See clause 8
Chloride content, max.		0,025		See clause 9
Organic surface coating?	—	No	Yes	See clause 8
Residue on 45 µm sieve, max.	% (*m*/*m*)	0,05	Not applicable	ISO 787-18
Matter volatile at 105 °C, max.	% (*m*/*m*)	3	1	ISO 787-2
Loss on ignition, max.	% (*m*/*m*)	2,5	10	ISO 3262-1
pH value of aqueous suspension	—	3,6 to 4,5	3,4 to 8[b]	ISO 787-9

[a] The carbon content is also part of the loss on ignition.

[b] Use a 1:1 (*m*/*m*) mixture of water and methanol.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Apparent density after tamping	g/ml	To be agreed between the interested parties		ISO 787-11
Specific surface area	m^2/g			To be agreed between the interested parties

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

6 Determination of silica content

6.1 Principle

A test portion is ignited, weighed and treated with sulfuric acid and hydrofluoric acid. The silicon tetrafluoride thus formed is evaporated off and the silica content is calculated from the resulting loss of mass.

As the silica content is very high, a previous separation from the impurities is not required.

6.2 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

6.2.1 Sulfuric acid, diluted 1 + 1.

Add 1 part by volume of concentrated sulfuric acid, approximately 96 % (*m*/*m*), $\rho \approx 1{,}84$ g/ml, slowly to 1 part by volume of water.

6.2.2 Hydrofluoric acid, concentrated, approximately 40 % (*m*/*m*), $\rho \approx 1{,}13$ g/ml.

6.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

6.3.1 Platinum dish.

6.3.2 Muffle furnace, capable of being maintained at (1 000 ± 20) °C.

6.3.3 Infrared evaporator.

6.3.4 Desiccator, containing magnesium perchlorate as desiccant.

6.4 Procedure

6.4.1 Number of determinations

Carry out the determination in duplicate.

6.4.2 Test portion

Weigh, to the nearest 0,2 mg, approximately 1 g (m_0) of the sample (see clause 5) into the tared platinum dish (6.3.1).

6.4.3 Determination

Ignite the test portion in the muffle furnace (6.3.2) at (1 000 ± 20) °C to constant mass (this should take approximately 2 h) and allow to cool in the desiccator (6.3.4). Weigh the test portion again (m_1).

Wet the ignited test portion in the platinum dish with 2 ml to 3 ml of water, add 1 ml of sulfuric acid (6.2.1) and 15 ml of hydrofluoric acid (6.2.2) and evaporate to a syrup on the infrared evaporator (6.3.3), taking care to avoid loss by spitting. Allow to cool and wash the sides down with small quantities of water. Then add a further 10 ml of hydrofluoric acid and evaporate to dryness. If the evaporation of the silicon tetrafluoride is not complete, add a further 10 ml of hydrofluoric acid and evaporate to dryness again.

Heat the residue until white fumes are no longer evolved, then ignite for 30 min in the muffle furnace at (1 000 ± 20) °C. Remove from the furnace, allow to cool in the desiccator and weigh to the nearest 0,2 mg (m_2).

6.5 Expression of results

Calculate the silica content $w(\mathrm{SiO_2})$, expressed as a percentage by mass, using the equation

$$w(\mathrm{SiO_2}) = \frac{(m_1 - m_2)}{m_1} \times 100$$

where

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

m_2 is the mass, in grams, after treatment with hydrofluoric acid and ignition.

Calculate the mean of the two determinations and report the result to the nearest 0,1 %.

6.6 Precision

No precision data are currently available.

7 Determination of aluminium oxide, titanium(IV) oxide and iron(III) oxide contents by spectrometry

7.1 Principle

A test portion is treated with sulfuric acid and hydrofluoric acid in a platinum dish. The resulting silicon tetrafluoride is volatilized and the residue is dissolved in hydrochloric acid. After diluting with water to a constant, known volume, the Al, Ti and Fe impurities are determined either by flame atomic absorption spectrometry (FAAS) or by inductively coupled plasma atomic emission spectrometry (ICP-AES), depending on which instrument is available in the laboratory.

NOTE The advantages of the ICP-AES method include its wide dynamic range and multi-element capabilities. Both methods (FAAS and ICP-AES) are relative analytical techniques. For quantitative analytical results, both measurement techniques must be calibrated using standard matching solutions.

7.2 Reagents and materials

Use only reagents of recognized analytical grade except for acids, which shall be ultrapure, and use only water of at least grade 3 purity as defined in ISO 3696.

7.2.1 Sulfuric acid, diluted 1 + 1.

Add 1 part by volume of concentrated sulfuric acid, approximately 96 % (*m*/*m*), $\rho \approx$ 1,84 g/ml, slowly to 1 part by volume of water.

7.2.2 Hydrofluoric acid, concentrated, approximately 40 % (*m*/*m*), $\rho \approx$ 1,13 g/ml.

7.2.3 Hydrochloric acid, concentrated, approximately 30 % (*m*/*m*), $\rho \approx$ 1,15 g/ml.

7.2.4 Hydrochloric acid, dilute, approximately 3 % (*m*/*m*), $\rho \approx$ 1,01 g/ml.

7.2.5 Caesium chloride buffer solution.

Dissolve 50 g of caesium chloride in approximately 500 ml of water and add 50 ml of concentrated hydrochloric acid (7.2.3). Make up to 1 000 ml with water and mix well.

7.2.6 Standard stock solutions, containing 1,000 g/l of aluminium, titanium and iron, respectively.

Store each solution in a fluorinated-polyethylene/polypropylene (FEP) bottle.

7.2.7 Standard solutions, containing 10 mg of the element per litre.

Prepare these solutions on the day of use.

Pipette 1 ml of the appropriate standard stock solution (see 7.2.6) into a 100 ml one-mark volumetric flask, add 10 ml of concentrated hydrochloric acid (7.2.3), make up to the mark with water and mix well.

1 ml of the standard solution contains 10 µg of the element concerned.

Prepare a more dilute or more concentrated standard solution, if necessary, depending on the concentration of Al, Ti or Fe in the product under test.

7.2.8 Ethanol, 96 % (*V*/*V*).

7.2.9 Acetylene (C_2H_2), commercial grade, in a steel cylinder.

7.2.10 Compressed air.

7.2.11 Dinitrogen oxide (N_2O), commercial grade, in a steel cylinder.

7.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

7.3.1 Flame atomic absorption spectrometer (FAAS), suitable for measurements at the following wavelengths:

- 309,3 nm for aluminium (Al),
- 364,3 nm for titanium (Ti),
- 248,3 nm for iron (Fe),

fitted with a suitable burner fed with

- an N_2O/C_2H_2 mixture for Al and Ti determinations,
- a C_2H_2/air mixture for Fe determinations,

and also fitted with hollow-cathode lamps for the elements Al, Ti and Fe and a deuterium background corrector.

7.3.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES), preferably with high resolution ($\leqslant$ 0,01 nm), automatic control of all plasma operating functions and a computer-controlled signal compensation system.

7.3.3 Platinum dish.

7.3.4 100 ml one-mark volumetric flasks, complying with the requirements of ISO 1042.

7.3.5 Pipettes, calibrated, of suitable volumes (see 7.4), complying with the requirements of ISO 648.

7.4 Procedure

7.4.1 Preparation of standard matching solutions

Introduce, with pipettes (7.3.5), suitable volumes of the standard solutions (7.2.7) into a series of several 100 ml one-mark volumetric flasks (7.3.4), add 10 ml of concentrated hydrochloric acid (7.2.3), make up to the mark with water and mix well. For the aluminium and titanium determination by FAAS, add 5 ml of caesium chloride solution (7.2.5) before making up to 100 ml with water.

7.4.2 Spectrometric measurement

7.4.2.1 Spectrometric measurement using FAAS

Install the required burner. Adjust the flow of C_2H_2 (7.2.9) and N_2O (7.2.11) or compressed air (7.2.10) (see Table 3) to suit the characteristics of the burner, and light the burner. Install the spectral source in the spectrometer (7.3.1) and optimize the conditions for the determination of the relevant element (Al, Ti or Fe). Adjust the instrument in accordance with the manufacturer's instructions in order to obtain the maximum absorbance. The test parameters are listed in Table 3.

Table 3 — FAAS test parameters

Element	**Spectral resonance line** nm	**Flame**	**Measurement range** µg of oxide/ml
Al	309,3	C_2H_2/N_2O	2 to 80
Ti	364,3	C_2H_2/N_2O	3 to 60
Fe	248,3	C_2H_2/air	0,2 to 6

Set the scale expansion, if fitted, so that the standard matching solution with the highest concentration gives an absorbance of about 0,55.

Aspirate into the flame each of the standard matching solutions in ascending order of concentration, and repeat with the solution of lowest concentration to verify that the instrument is stable.

Aspirate water into the flame after each measurement.

7.4.2.2 Spectrometric measurement using ICP-AES

Introduce the standard matching solutions into the plasma using an air atomizer, having set the instrument to the manufacturer's recommended operating conditions and using spectral resonance lines as given in Table 4.

Table 4 — ICP-AES parameters

Element	**Spectral resonance line** nm	**Measurement range** µg of oxide/ml
Al	309,27	0,2 to 20
Ti	334,94	
Fe	238,20	0,1 to 10

7.4.3 Calibration graph

Correct each absorbance by subtracting the reading obtained from the blank matching solution and plot for each element a graph having the masses, in micrograms, of the element contained in 1 ml of standard matching solution as abscissae and the corresponding values of the absorbance as ordinates.

7.4.4 Preparation of the test solution

Carry out the determinations in duplicate.

Weigh, to the nearest 1 mg, approximately 5 g (m_0) of the sample (see clause 5) into the platinum dish (7.3.3). Wet this test portion with 5 ml of water for grade A fumed silica and with 3 ml to 4 ml of ethanol (7.2.8) and 2 ml to 3 ml of water for grade B fumed silica.

Add 2 ml of sulfuric acid (7.2.1) and 30 ml of hydrofluoric acid (7.2.2) and evaporate carefully to a syrup. Then add a further 10 ml of hydrofluoric acid and evaporate to dryness. If the evaporation of the silicon tetrafluoride is not complete, add a further 10 ml of hydrofluoric acid and evaporate again.

Heat the residue until white fumes are no longer evolved, then add 5 ml of hydrochloric acid (7.2.3) and 2 ml to 3 ml of water and dissolve the residue.

Transfer the solution obtained to a 50 ml one-mark volumetric flask. For aluminium and titanium determinations by FAAS, add 2,5 ml of caesium chloride buffer solution (7.2.5). Then make up to 50 ml with water.

7.4.5 Determination of aluminium, titanium and iron

First adjust the spectrometer as described in 7.4.2.1 or 7.4.2.2 and measure the absorbance of the blank matching solution (see 7.4.1) in the spectrometer (7.3.1 or 7.3.2). Then measure the absorbance of the test solution (see 7.4.4) three times and, afterwards, that of the blank matching solution again. Finally, redetermine the absorbance of the standard matching solution with the highest concentration in order to verify that the response of the apparatus has not changed.

If the absorbance of the test solution is higher than the absorbance of the standard matching solution with the highest concentration, dilute the test solution with a known volume of dilute hydrochloric acid (7.2.4). Note the dilution factor D.

7.4.6 Determination of the total loss on ignition

Carry out the determination in duplicate.

Weigh, to the nearest 0,2 mg, approximately 1 g (m_3) of the sample (see clause 5) into a platinum crucible (6.3.1).

NOTE Weighing out the test portions for the determination of the silica content (see 6.4.2) and the total loss on ignition may be carried out at the same time.

Ignite the test portion to constant mass in a muffle furnace (6.3.2) (this should take approximately 2 h) at (1 000 ± 20) °C and allow to cool in a desiccator (6.3.4). Weigh the ignited test portion to the nearest 0,2 mg (m_4).

Calculate the total loss on ignition $w(\text{TLI})$, expressed as a percentage by mass, using the equation

$$w(\text{TLI}) = \frac{m_3 - m_4}{m_3} \times 100$$

where

m_3 is the mass, in grams, of the test portion before ignition;

m_4 is the mass, in grams, of the ignited test portion.

Calculate the mean of the two determinations and report the result to the nearest 0,001 %.

7.5 Expression of results

7.5.1 Calculation of the concentration of each element

Calculate the concentration c_t, expressed in micrograms per millilitre, of each element (Al, Ti, Fe) in the test solution (see 7.4.4), using the equation

$$c_\mathrm{t} = \frac{c_\mathrm{c} \times A_\mathrm{t}}{A_\mathrm{c}}$$

where

c_c is the concentration, in micrograms per millilitre, of the element in the standard matching solution;

A_t is the absorbance (or extinction) value for the test solution;

A_c is the absorbance (or extinction) value for the standard matching solution.

7.5.2 Calculation of the oxide content for each element

Calculate the content of each oxide (Al_2O_3, TiO_2, Fe_2O_3), w(oxide), expressed as a percentage by mass, using the equation

$$w(\text{oxide}) = \frac{c_\mathrm{t} \times V_0 \times D \times f}{m_0 \left[1 - \dfrac{w(\text{TLI})}{100}\right]} \times 100$$

where

c_t is the concentration, in micrograms per millilitre, of the element in the test solution;

V_0 is the volume, in millilitres, of the test solution (50 ml);

D is the dilution factor (if applicable, see 7.4.5);

f is a conversion factor, for calculation of the oxide content rather than the content of the element, as given in Table 5;

m_0 is the mass, in grams, of the test portion;

w(TLI) is the total loss on ignition as determined in 7.4.6.

Calculate the mean of the two determinations and report the result to the nearest 0,001 % (for Al_2O_3 and TiO_2) or 0,000 1 % (for Fe_2O_3).

Table 5 — Values of conversion factor *f*

Element	Oxide	Conversion factor
Al	Al_2O_3	1,889 5
Ti	TiO_2	1,668 1
Fe	Fe_2O_3	1,429 8

7.6 Precision

No precision data are currently available.

8 Determination of carbon content

8.1 Principle

A test portion in a crucible is covered, if necessary, with a suitable catalyst, and combusted in a stream of oxygen in an induction furnace.

Sulfur compounds, halogens and water vapour are removed from the combustion products, which are then passed over a platinum catalyst (to convert carbon monoxide to carbon dioxide), and the carbon dioxide concentration is measured using an infrared-cell detector.

Alternatively, the carbon can be determined by conductivity measurement. In this case, the specified combustion products are passed over a platinum catalyst and the carbon dioxide present is absorbed in a sodium hydroxide (NaOH) solution. The change in conductivity of the solution (caused by the conversion of some of the NaOH to disodium carbonate, Na_2CO_3) is measured.

8.2 Reagents and materials

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

8.2.1 Oxygen, purity 99,99 % min.

8.2.2 Catalyst, comprising iron chips plus a mixture consisting of 9 parts by mass of tungsten and 1 part by mass of tin powder.2)

NOTE The catalyst is used as required to obtain satisfactory results.

8.2.3 Carbon reference materials (carbon steels).3)

8.2.4 Platinum catalyst pellets, suitable for use at 400 °C to 450 °C, to convert carbon monoxide to carbon dioxide.

8.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

8.3.1 Low-carbon analyser, consisting of an induction-heated furnace suitable for operation at about 1 800 °C, a scavenging unit, a platinum catalyst system operating at about 450 °C, and an infrared-cell detection system. Alternatively, a carbon dioxide absorption unit including an NaOH solution and equipment for measuring the change in conductivity can be used.

The system shall include a carbon dioxide absorbent based on NaOH, a moisture absorbent (magnesium perchlorate) for purification purposes and a flowmeter for control of the oxygen stream.

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2) For example Lecocel II®.

3) Suitable examples are:

NIST SRM 131b (containing 0,001 8 % C); BAM 238-1(containing 0,018 % C); NIST SRM 151 (containing 0,55 % C); BAM EURO-ZRM 476-2 (containing 3,43 % C).

8.3.2 Crucibles, expendable, made of alumina or similar refractory material. Both crucible and lid shall be ignited before use at a temperature of 1 000 °C or higher for a time (usually 20 min) sufficient to give a constant mass.

8.4 Procedure

8.4.1 Preparation of apparatus

Follow the operating instructions for the specific equipment used. After setting the controls, carry out several blank runs with a crucible (8.3.2) containing the required amount of catalyst but not the test portion. Successive blank values shall approach a low, constant value.

8.4.2 Calibration

Weigh, to the nearest 0,1 mg, approximately 0,5 g of reference material (8.2.3) into a crucible, combust and record the result if using equipment which gives the result automatically as described in 8.4.3, or use the equation given in 8.5 if using equipment which does not. Repeat at least twice. Adjust the calibration controls to produce the correct readings on the direct-reading meter. Combust additional samples of the reference material as required to produce the correct direct reading.

8.4.3 Determination

Carry out the determination in duplicate.

Weigh, to the nearest 0,1 mg, approximately 0,5 g of the sample (clause 5) (m_0) into a crucible, cover with 0,7 g of iron chips and 1,0 g of tungsten/tin powder (8.2.2) and place the crucible in the induction furnace of the low-carbon analyser (8.3.1). It is especially important to cover the test portion completely with the catalyst when the test portion is a powder as this prevents possible blow-out of test portion during ignition.

If the analyser has an integral balance, the test portion mass is automatically stored in the memory. In this case, press the "analyse" key and the analysis proceeds automatically. The result is displayed on the screen and is printed as percent by mass of carbon in the test portion.

8.5 Expression of results

If the equipment used does not print the result automatically, calculate the carbon content $w(\mathrm{C})$, expressed as a percentage by mass, using the equation

$$w(\mathrm{C}) = \frac{m_\mathrm{t} - m_\mathrm{b}}{m_0 \left[1 - \dfrac{w(\mathrm{TLI})}{100} \right]} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_b is the mass, in grams, of carbon recorded in the final blank determination;

m_t is the mass, in grams, of carbon in the test portion;

$w(\mathrm{TLI})$ is the total loss on ignition as determined in 7.4.6.

Calculate the mean of the two determinations and report the result to the nearest 0,01 %.

8.6 Precision

No precision data are currently available.

9 Determination of chloride content

9.1 Principle

A test portion of fumed silica is dissolved in sodium hydroxide solution. The chloride content is determined by potentiometric titration against silver nitrate solution.

9.2 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

9.2.1 Sodium hydroxide, suprapure, 20 % (m/m) solution.

9.2.2 Nitric acid, suprapure, diluted 1 + 1.

Add 1 part by volume of concentrated nitric acid, approximately 65 % (m/m), ρ = 1,40 g/ml, to 1 part by volume of water.

9.2.3 Standard silver nitrate solution, $c(AgNO_3)$ = 0,1 mol/l or 0,01 mol/l, prepared from analytical-grade silver nitrate.

NOTE The solution used will depend on the chloride content of the sample.

9.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

9.3.1 Potentiometer, having an input resistance $\geqslant 10^{13}\ \Omega$ and a resolution $\geqslant$ 0,1 mV.

9.3.2 Reference electrode, for example a standard calomel electrode, and **measurement electrode,** for example a chloride-ion-selective electrode or silver electrode.

9.3.3 150 ml beakers, high form, complying with the requirements of ISO 3819.

9.3.4 Ice bath.

9.3.5 Magnetic stirrer, including a stirrer bar coated with polytetrafluoroethylene.

9.3.6 Thermometer, capable of being used between – 5 °C and 30 °C.

9.4 Procedure

9.4.1 Number of determinations

Carry out the determination in duplicate.

9.4.2 Test portion

Weigh, to the nearest 1 mg, between 1 g and 5 g (m_0) of the sample (see clause 5), depending on the chloride content of the sample, into a beaker (9.3.3).

9.4.3 Determination

Dissolve the test portion in 20 ml of sodium hydroxide solution (9.2.1) by gently warming. Rinse the sides of the beaker with water.

Introduce 15 ml of nitric acid (9.2.2) into a second beaker (9.3.3). Cool both solutions to about 0 °C by placing them in the ice bath (9.3.4) on the magnetic stirrer (9.3.5).

Slowly add the sodium silicate solution obtained by dissolving the test portion to the nitric acid in the second beaker, keeping the beaker containing the nitric acid in the ice bath and stirring continuously. During the addition, the temperature of the mixture, as indicated by the thermometer (9.3.6), shall not exceed 20 °C.

WARNING — Never add the nitric acid to the sodium silicate solution.

Titrate the acidic test solution thus obtained with one of the standard silver nitrate solutions (9.2.3) (depending on the chloride content) at room temperature, using the electrodes (9.3.2) and the potentiometer (9.3.1) to detect the end-point.

Note the volume (V_1) of silver nitrate solution required to reach the end-point.

Carry out a blank determination, using the same procedure and the same reagents.

9.5 Expression of results

Calculate the chloride content $w(\text{Cl})$, expressed as a percentage by mass, using the equation

$$w(\text{Cl}) = \frac{(V_1 - V_b) \times c(\text{Ag}) \times f}{m_0 \left[1 - \frac{w(\text{TLI})}{100}\right]} \times 100$$

where

V_1 is the volume, in millilitres, of silver nitrate solution (9.2.3) required by the test solution;

V_b is the volume, in millilitres, of silver nitrate solution (9.2.3) required for the blank determination;

$c(\text{Ag})$ is the concentration, in millimoles per millilitre, of the silver nitrate solution (9.2.3);

f is the chloride factor, in milliequivalents ($f = 0{,}035\,43$);

m_0 is the mass, in grams, of the test portion;

$w(\text{TLI})$ is the total loss on ignition, as determined in 7.4.6.

Calculate the mean of the two determinations and report the result to the nearest 0,000 1 %.

9.6 Precision

No precision data are currently available.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this part of ISO 3262 (ISO 3262-20);

c) the results of the tests and whether or not the product complies with the relevant specification limits;

d) any deviation from the test methods specified;

e) the dates of the tests.

ICS 87.060.10

Price based on 13 pages