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BS 1795:1976 ISO 3262:1975

Specification for

Extenders for paints

UDC 667.622.52



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Foreword

This British Standard for extenders for paints was first published in one volume in 1952 and subsequently revised in 1965.

This revision brings the British Standard into line with ISO 3262:1975, *"Extenders for paints"*.

For the purposes of this British Standard the text of ISO 3262 given in this publication should be modified as follows.

Terminology. The words "British Standard" should replace "International Standard" wherever they appear. The decimal point should replace the decimal comma wherever it appears.

Cross-references. The references to other International Standards should be replaced by references to British Standards as follows:

Clause	Reference to ISO Standard	Appropriate British Standard
Table 1	ISO 787-XVIII	BS 3483 "Methods for testing pigments for paints" Part B4 "Determination of residue on sieve (water method, using a mechanical flushing procedure")
6	ISO 842	BS 4726 "Methods for sampling raw materials for paints and varnishes"
8	ISO 48	BS 903 "Methods of testing vulcanized rubber" Part B6 "Total sulphur" (included in BS 903-B6 to BS 903-B10)
10	ISO 787-II	BS 3483-B6 "Determination of matter volatile at 105 °C"
12.2	ISO 787-III	BS 3483-C1 "Determination of matter soluble in water (hot extraction method)"
13	ISO 787-IX	BS 3483-C4 "Determination of pH value of aqueous suspension"
Annex	ISO 787-V	BS 3483-B7 "Determination of oil absorption value"

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope and field of application

This International Standard specifies the principal requirements and the corresponding methods of test for certain paint extenders.

It is applicable to the types and grades of paint extender listed in clause 4.

2 References

ISO 787, General methods of test for pigments. ISO 842, Raw materials for paints and varnishes — Sampling.

3 Definition

extender

an inorganic material in powder form, with a refractive index usually less than 1,7, used as a constituent of paints to confer special effects and to modify properties such as sedimentation, working and film-forming properties

4 Types and grades

The extenders are classified in types according to the nature of the material, and in grades according to their residue on sieve and particle size.

Extender No. 1: Barytes (grades a, b)

Barium sulphate, natural.

Extender No. 2: Blanc fixe

Barium sulphate, precipitated.

Extender No. 3: Whiting (grades a, b, c, d)

Calcium carbonate, natural. Craie, Kreide. Naturally occurring cretaceous chalk, primarily $CaCO_3$ of micro-crystalline form (essentially remains of coccoliths and foraminifera).

Extender No. 4: Calcium carbonate, crystalline (grades a, b, c, d)

Limestone, Iceland spar, natural calcium carbonate other than that covered by extender No. 3.

Extender No. 5: Calcium carbonate, precipitated (grades a, b)

Calcium carbonate prepared by precipitation.

Extender No. 6: Dolomite (grades a, b, c, d)

Calcium magnesium carbonate. In chemical composition, approximately equimolecular proportions of calcium and magnesium carbonates.

Extender No. 7: China clay (grades a, b, c)

Aluminium silicate, natural, hydrated. Kaolin, essentially kaolinite, $Al_2O_3.2SiO_2.2H_2O$, of lamellar crystalline form.

Extender No. 8: Talc, fibrous form (grades a, b)

Magnesium silicate, natural, hydrated, $3MgO.4SiO_2$.H₂O.

Extender No. 9: Talc, lamellar form (grades a, b, c)

Magnesium silicate, natural, hydrated, $3MgO.4SiO_2.H_2O.$

Extender No. 10: Talc, containing carbonate

Magnesium silicate, natural, hydrated, $3MgO.4SiO_2 \cdot H_2O$, lamellar form. Different from extender No. 9 in that it contains carbonate.

Extender No. 11: Mica (grades a, b)

Potassium aluminium silicate, natural, hydrated, $K_2O.3Al_2O_3.6SiO_2.2H_2O$, lamellar form. Muscovite. Wet ground (grade a) and dry ground (grade b).

Extender No. 12: Silica (quartzite), type 1 (grades a, b, c, d)

Silica, natural. SiO_2 content not less than 80 % but less than 98 %.

Extender No. 13: Silica (quartzite), type 2 (grades a, b, c, d)
Silica, natural, ${ m SiO}_2$ content equal to or greater than 98 %.
Extender No. 14: Kieselguhr, type 1 (grades a, b, c, d)
Diatomaceous siliceous earth. ${ m SiO}_2$ content not less than 70 % but less than 80 %.
Extender No. 15: Kieselguhr, type 2 (grades a, b, c, d)
Diatomaceous siliceous earth. SiO_2 content not less than 80 %.
Extender No. 16: Calcium silicate, synthetic
Hydrated synthetic calcium silicate.
Extender No. 17: Sodium aluminium silicate, synthetic
Hydrated synthetic sodium aluminium silicate.
Extender No. 18: Silica, synthetic
Hydrated synthetic silica.

5 Composition and properties

The material shall be in the form of a dry powder or in such condition that it may readily be reduced thereto by crushing under a palette knife without any grinding action being necessary. When examined microscopically at a suitable magnification, it shall closely resemble an agreed sample.

The composition and properties of the materials shall be as given in Table 1.

It should be noted, however, that in Table 1 no reference is made to oil-absorption values; these are given in the table in the Annex for information only.

6 Sampling

Sampling shall be carried out in accordance with ISO 842.

Methods of test

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of equal purity shall be used throughout the tests.

7 Comparison of colour

NOTE There is no objection to the use of a particular instrumental method, agreed between the interested parties, for comparison of colour. However, in view of differences between the results obtained with various instruments, the simple visual method is given as the reference method.

7.1 Procedure

Place approximately equal masses of the test sample and the agreed sample on a ground glass plate placed on a piece of white paper.

Place a piece of thin glass plate vertically between the two samples so as to form a partition. Push the samples towards each other until they come into contact with the glass plate. Remove the glass plate and flatten the surface of the two heaps with another glass plate which shall then be removed before viewing.

Examine the colour of the samples in diffuse daylight or, if good daylight is not available, make the comparison in artificial daylight.

Add carefully a few drops of either white spirit or water (as agreed between the interested parties) in such a way that the two samples are just wetted without being disturbed, and repeat the colour comparison by the same method as before.

8 Determination of residue on sieve

8.1 Apparatus

 $\mathbf{2}$

8.1.1 Three sieves, 100 to 200 mm diameter, with sides of height 50 mm and meshes of apertures 45, 63 and $125 \,\mu$ m respectively.

8.1.2 *Glass rod*, tipped with rubber having a hardness of 50 IRHD^{1} .

8.1.3 *Filter crucible of sintered glass,* pore size index P 40 (diameter of pores 16 to 40 μ m).

¹⁾ See ISO 48, Vulcanized rubbers — Determination of hardness.

			1			idue on si		Pa	rticle size		on ^b	Matter volatile at 105 °C	Loss on ignition	Matter soluble in water	
	Extender	Composition % (m/m)	Test method	Colour (see clause 7)		ee clause (<i>m/m</i>) ma			(see cl % (m/n			(see clause 10) % (m/m) max.	(see clause 11) % (m/m)	(see clause 12) % (m/m)	pH of aqueous suspension (see clause 13)
No.					$125\mu{\rm m}$	$63\mu{ m m}$	$45\mu{ m m}$	$< 20 \mu { m m}$	$< 10 \mu m$	$< 5 \mu{ m m}$	$< 2 \mu$ l m ^c			max.	
1	Barytes grade a grade b	BaSO ₄ , 90 min.	see clause 14				2,0 0,5	65 99	40 95	20 80	(5) (20)	0,5	0,5 max.	0,5	6 to 10
2	Blanc fixe	BaSO ₄ , 95 min.	see clause 14		0,02	0,1	0,2	90	80	60	(25)	0,5	_	0.5 ^d	5,5 to 9,5
3	Whiting											- / -		0,5	-,,-
	grade a grade b grade c grade d	CaCO ₃ , 96 min. 99 max.	see clause 15	le,	0,5 0,01 —	6 0,5 0,05 —	$ \begin{array}{c} 15 \\ 2 \\ 0,5 \\ 0,01 \end{array} $			$\frac{-}{45}$ 55 70	(20) (25) (35)	0,3	42 to 44	0,15	8,0 to 9,5
4	Calcium carbonate (crystalline) grade a grade b	CaCO ₃ , 98 min.	see clause 16	No extender to be darker or more strongly coloured than an agreed sample	0,01 0,01	1,0 0,5 0,01	5,0 1,5 0,1	70 80 95	40 45 65 98	25 30 35	(10) (10) (15)	0,3	42 to 44.5	0,15	8 to 10
	grade c grade d	~		an a	_		$0,1 \\ 0,01$	95	65 98	35 70	(15) (25)	0,5	42 10 44,5	0,10	
5	Calcium carbonate (precipitated)			d than											1
	grade a grade b	CaCO ₃ , 97 min. 100 max.	see clause 15	oure	0,1 0,1	$^{0,25}_{0,25}$	0,5 0,5	90	70 90	40 70	(20)	} 1,0	} 43,5 to 44,5	} 0,3	} 8 to 10,5
6	Dolomite			/ col											
	grade a grade b grade c grade d	CaMg(CO ₃) ₂ , 97 min. ^e	see clause 16	re strongly	0,01 0,01 —	1,0 0,5 0,01 —	5,0 1,5 0,1 0,01	70 80 95 —	40 45 65 98	25 30 35 70	(10) (10) (15) (25)	0,3	46 to 48	0,2	8 to 10,5
7	Chine clay			, mo											1
	grade a grade b grade c			arker on			$^{0,5}_{0,1}_{0,05}$	90 95 99,5	70 80 99		(15) (35) (70)	2	} 10 to 14	} 0,5	} 4,5 to 9,5
8	Talc, fibrous form grade a grade b			r to be d	_	_	$1,5 \\ 0,5$	75 90	55 75	40 50	(20) (25)	0,5	} 4 to 7	} 0,5	8,5 to 10
9	Talc, lamellar form			ndeı	_	_	0,5	50	10	50	(23)	J	J	J	J
	grade a grade b grade c			-No exte	0,01 	$0,5 \\ 0,01 \\ -$	$^{3,0}_{0,10}_{0,01}$	80 95 —	50 70 95	30 40 70	(8) (15) (25)	0,5	} 4 to 8	0,5	8,5 to 10
10	Talc, containing carbonate											,			
	grade a grade b grade c	Magnesium silicate with a max. of 50 % MgCO ₃ + CaCO ₃	see clause 16		0,01 	$0.5 \\ 0.01 \\ -$	${}^{3,0}_{0,10}_{0,01}$	80 95 —	50 70 95	30 40 70	$(8) \\ (15) \\ (25)$	} 0,5	} 28 max.	$\left. ight\} = 0,5$	} 8 to 10,5
11	Mica grade a grade b				_	1,0	3,0 0,5	98 98	80 70	30 30	(5) (10)	0,5	} 5,5 max.	} 0,5	7 to 9,5

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Table 1 — Composition and properties of extenders

Table 1 — Composition and properties of extenders															
No.	Extender	Composition % (m/m)	Test method	Colour (see clause 7)	(s	idue on si ee clause 5 (m/m) ma 63 μm	8)	Pa < 20 μm		ause 9) n) min.	on ^b $< 2 \mu m^{c}$	Matter volatile at 105 °C (see clause 10) % (m/m) max.	Loss on ignition (see clause 11) % (m/m)	Matter soluble in water (see clause 12) % (m/m) max.	pH of aqueous suspension (see clause 13)
12	Silica (quartzite) type 1 grade a grade b grade c grade d	${\rm SiO}_2,80$ to 98	pq	agreed sample.—	40 2 —	 25 0,5 	-60 5,0 0,1	 70 		 10 95		0,5	5 max.	0,5	7 to 10
13	Silica (quartzite) type 2 grade a grade b grade c grade d	SiO ₂ , 98 min.	classical method	coloured than an agr	40 2 		$\overline{\begin{array}{c} 60\\ 5,0\\ 0,1 \end{array}}$	 70 	 	 10 95		0,5	0,2 max.	0,5	7 to 8,5
14	grade c grade d	${ m SiO}_2,70$ to 80	Any recognized	more strongly colour	 			70 75 80 90	10 20 30 50	5 10 30	 	12	> 15 max.	} 1,0	6,5 to 10
15	grade d	${ m SiO}_2,$ 80 min.		arker or	 			70 75 80 90	10 20 30 50	$\frac{-}{5}$ 10 30		2	1 max.	1,0	6,5 to 10
16		To be agreed between the parties	ne interested	be da	—	1,0	3,0	90	70	40	_	12	8 max.		8 to 12,5
17	Sodium aluminium silicate, synthetic			der to	_	2,0	4,0	85	50	20	_	12	8 max.	To be agreed	8 to 12,5
18	Silica, synthetic	${ m SiO}_2,$ 97 min.	see clause 17	—No extender		2,0	2,0	80	50	20	_	12	8 max.	between the interested parties	3,5 to 8,0

Table 1 — Composition and properties of extenders

^a The determination of residue on sieve using the method in this International Standard requires considerable experience. More reproducible results may be obtained if a mechanical flushing method is used (See ISO 787-XVIII) but these results will be lower than those obtained using the manual method. The results obtained, therefore, by the different methods should not be used for comparison.

^b The method for the determination of particle size distribution is based on Stokes' law. Strictly speaking, Strokes' law applies to spherical particles. Extender particles are not strictly spherical, therefore the given figures are not an absolute measure of particle size, but serve as a means of comparing different samples of the same extender. They should not be used to compare one extender with another because different extenders have different particle shapes. Furthermore, the figures do not necessarily relate to those given under "Residue on sieve", since these refer to actual dimensions of the particles.

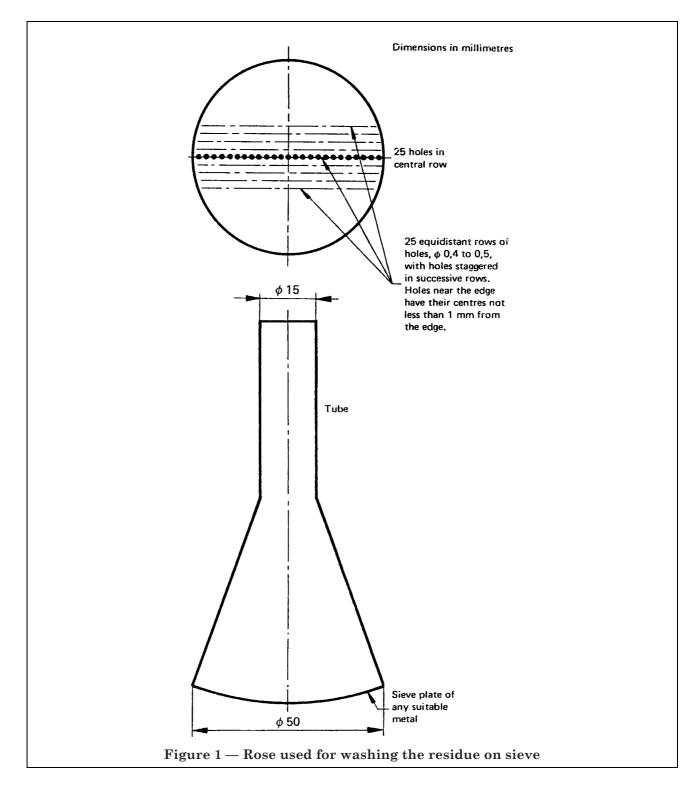
² The 2 μ m limits are given for guidance only.

^d Excluding water-soluble alkaline earth compounds. (See **12.3**, Method 1A.)

^e The proportion by mass of CaCO₃ to MgCO₃ is in practice between 1,18 and 1,23 CaCO₃ : 1,0 MgCO₃.

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8.1.4 *Water supply*, at a pressure equivalent to a head of water of 1 m.

8.1.5 Rose, in accordance with Figure 1.

8.2 Procedure

Weigh into a beaker, to the nearest 0,1 g, between 50 and 500 g (see note 1) of the test sample previously dried in accordance with clause **10**. Disperse the extender (see note 2) in a suitable quantity of water (usually of the order of 500 to 600 ml), using the rubber-tipped glass rod (**8.1.2**) only to assist in the dispersion, and pour the dispersion through the appropriate sieve (**8.1.1**). Return the residue retained on the sieve to the beaker and repeat the dispersion (without adding any further dispersing agent), using a similar quantity of water; pour onto the sieve as before. Repeat this operation once more and wash out the residue remaining in the beaker with water, using the glass rod if necessary.

Wash the residue on the sieve, using the rose (8.1.5) fitted to the constant-head water supply (8.1.4) until the washings are clear.

Wash the residue into the previously tared sintered glass crucible (8.1.3) and dry at 105 ± 2 °C. Cool in a desiccator and weigh to the nearest 0,1 mg. Repeat the heating, cooling and weighing operations until constant mass is obtained.

NOTE 1 The mass of extender chosen shall be such that, wherever possible, the expected sieve residue is about 0,2 g. With the finest extenders, samples up to 500 g shall be used.

NOTE 2 If necessary, a suitable wetting or dispersing agent may be used, such as ethanol or a condensed naphthalene sulphonate.

8.3 Expression of results

The residue on sieve, as a percentage by mass, is given by the formula:

 $\underline{100\ m_1}$

 m_0

where

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

 m_1 is the mass, in grams, of the residue.

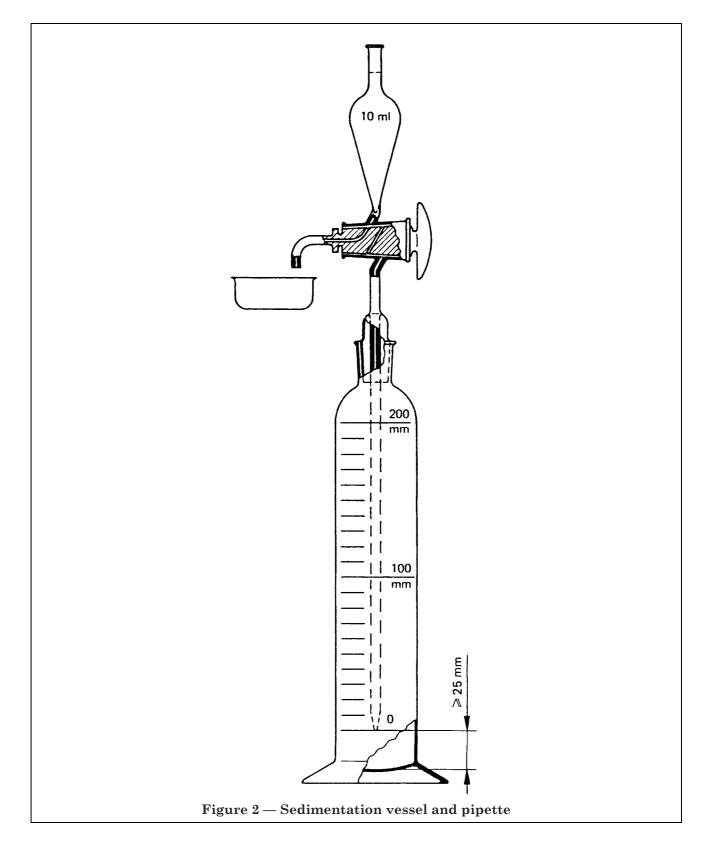
9 Determination of particle size distribution

NOTE Because of the ease of execution and the good reproducibility of the method, the Andreasen method is included as the reference method. Other methods may, however, be used by agreement between the interested parties, but in such cases it will be necessary also to agree on appropriate limits.

9.1 Principle

The rate of fall of spherical particles through a medium in which they are dispersed is proportional to the square of the particle diameter (Stokes' law). The Andreasen method for determination of particle size distribution makes use of this relationship and expresses the particle size distribution in terms of distribution of spherical particles of the same settlement rate. In the determination, a dilute suspension is prepared and the concentration of solids at a fixed point below the surface is determined at a series of time-intervals calculated to correspond to certain equivalent spherical diameters.

9.2 Apparatus (see Figure 2).



9.2.1 Sedimentation vessel, glass, about 56 mm internal diameter and having a graduated scale from 0 to 200 mm marked on its side. The zero graduation line shall be not less than 25 mm from the inside base of the vessel, and the capacity of the vessel up to the 200 mm line shall be about 550 to 620 ml.

9.2.2 *Pipette*, fitted with a two-way tap and side discharge tube. The capacity of the pipette to the graduation line is conveniently 10 ml. A bell-shaped dome with a ground glass joint to fit the neck of the sedimentation vessel is fused to the pipette. A small vent hole is made in this dome. The inlet to the pipette stem must be level with the zero line on the sedimentation vessel. The stem from the pipette bulb to the sampling inlet shall be constructed of capillary glass tubing with a bore not less than 1 mm and not more than 1,3 mm. The tube above the bulb shall be 4 to 4,5 mm bore.

9.2.3 Transparent-sided constant-temperature bath of at least 15 l capacity, maintained at a temperature of 23 ± 0.5 °C, into which the sedimentation vessel can be immersed upto the 200 mm graduation line. The bath shall be positioned away from sources of vibration and the circulating system shall not cause vibration.

9.2.4 Mechanical stirrer capable of rotating at a suitable speed for complete

dispersion (1 000 \pm 100 rev/min is generally suitable). The stirrer shall be designed to lift the dispersion and to avoid the creation of a vortex.

NOTE $\,$ A suitable stirrer may be made from an approximately 40 mm diameter brass disk with four equally spaced cuts, the cut sections being turned upwards at an angle of 30° to the horizontal.

9.2.5 Dispersion vessel of appropriate dimensions, such as a 1 000 ml gas jar.

9.2.6 Balance having a sensitivity of not less than 0,1 mg.

9.2.7 Drying oven capable of being maintained within a temperature range suitable for evaporation of the suspending liquid, for example 105 ± 2 °C for water.

9.2.8 Wide-mouthed weighing bottles suitable for evaporation, of capacity not less than 20 ml, or a *small* laboratory centrifuge and centrifuge tubes preferably of 20 ml capacity but of not less than 10 ml capacity. **9.2.9** Stop-watch or stop-clock

9.3 Preparation for the test

9.3.1 Calibration of pipette

Thoroughly clean the pipette (9.2.2). Partly fill the sedimentation vessel (9.2.1) with distilled water. Set the tap in the sampling position and, by means of a rubber tube, suck water into the bulb to the level of the graduation line. Reverse the tap to the discharge position and allow the water to drain into a tared weighing bottle (9.2.8). Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the weighing bottle. Weigh the bottle to the nearest 0,001 g and calculate from this mass the internal volume $V_{\rm p}$ of the pipette.

9.3.2 Calibration of sedimentation vessel

Thoroughly clean the sedimentation vessel, and weigh it, with the pipette in place, to the nearest 0,1 g. Then fill it to the 200 mm line with water at 23 ± 0.5 °C and reweigh. Calculate from the mass of water the internal volume $V_{\rm s}$ of the vessel.

9.3.3 Analysis sample

Using the quantities given in Table 2, prepare a dispersion by placing the test portion in the dispersion vessel (9.2.5), adding dispersion solution at 23 ± 0.5 °C to give a total volume of about 500 ml and stirring with the mechanical stirrer (9.2.4) for 15 min. Immediately pour all the suspension into the sedimentation vessel and make up to the 200 mm line with water or aqueous alcohol as appropriate. Transfer the sedimentation vessel to the constant-temperature bath (9.2.3).

9.4 Procedure

9.4.1 Sedimentation

Allow the sedimentation vessel to stand immersed to the 200 mm line until it has reached the temperature of the bath. Record this temperature, which shall not differ greatly from that of the room.

When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole and inverting the vessel several times. Immediately after mixing, replace the vessel and start the stop-clock or stop-watch (9.2.9).

Withdraw from the suspension by means of the pipette a series of samples of volume V_p at depths h_n at increasing time intervals after mixing, starting at time t_1 corresponding to a particle size of about 20 µm (calculated in accordance with **9.4.2**) and thereafter in progression so that the limiting diameters stand in a $\sqrt{2}$: 1 progression. Alternatively, the samples may be taken at convenient times approximately in a 2 : 1 progression and the exact spherical particle diameters corresponding to each sample calculated. The time to fill the pipette shall be about 20 s.

	Extender	Mass to be used (previously dried in accordance with clause 10) and weighed to an accuracy of 0,01 g	Mass and type of dispersing agent (solid active material)		
No.		g			
1	Barytes	20	0,75 g of sodium hexametaphosphate		
2	Blanc fixe	20	dissolved in 750 ml of water		
3	Whiting	3,5			
4	Calcium carbonate, crystalline	3,5	0,07 to $0,14$ g of suitable agent ¹⁾		
5	Calcium carbonate, precipitated	3,5	dissolved in 750 ml of water		
6	Dolomite	3,5			
7	China clay	12	0,2 g of sodium carbonate and 0,1 g of sodium hexametaphosphate dissolved in 750 ml of water		
8	Talc, fibrous form	10	0,40 g of sodium pyrophosphate		
9	Talc, lamellar form	10	dissolved in 750 ml of 20 % (m/m)		
10	Talc, containing carbonate	10	ethanol in water ²⁾		
11	Mica	15	1,5 g of sodium hexametaphosphate dissolved in 750 ml of water		
12 and 13	Silica (quartzite) type 1 and type 2				
14 and 15	Kieselguhr, type 1 and type 2		0,75 g of sodium hexametaphosphate		
16	Calcium silicate, synthetic	5	dissolved in 750 ml of water		
17	Sodium aluminium silicate, synthetic				
18	Silica, synthetic	4			

NOTE Freshly boiled distilled water or water otherwise prepared of equal purity shall be used when making up the dispersions.

Certain sodium salts of polymethacrylates have been found most suitable. Such materials are sold as Polysaltz, Dispex, etc.
 For reference purposes, 100 % ethanol shall be used.

Discharge the suspension from the pipette into a tared weighing bottle (9.2.8). When the bulb has drained, remove the rubber suction tube and run 5 to 7 ml of water from an ordinary 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface. The stem of the sedimentation pipette shall remain filled with suspension. Dry the contents of the weighing bottle at 105 °C until the difference between the successive weighings is not greater than 0,1 mg. Calculate the mass of the fraction, m_n , allowing for the mass of dispersing agent, which shall be determined by carrying out a blank test. The determination of the different fractions may be carried out by means of chemical methods if this procedure is more suitable.

The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for it in the subsequent calculations. Suppose that the decrease is 4 mm for each fraction withdrawn, and that initially the depth of immersion of the pipette was 200 mm, then the depth after the first fraction has been withdrawn will be 196 mm, and the mean depth h_1 used for calculation of the initial diameter corresponding to the first fraction will be 198 mm. The mean depth h_2 for the second fraction will be 194 mm, and so on.

$9.4.2 \ Calculation \ of \ with drawal \ time$

The withdrawal time, t_n , in seconds, for the *n*th sample is given by the equation

$$t_n = \frac{18\eta h_n}{(\rho_2 - \rho_1) gd_n^2} \times 10^6$$

where

- η is the absolute viscosity, in newton seconds per square metre, of the medium;
- h_n is the mean depth, in millimetres, at which the *n*th sample extraction is made;
- ho_1 is the density, in megagrams per cubic metre (or grams per cubic centimetre), of the medium;
- ρ_2 is the density, in megagrams per cubic metre (or grams per cubic centimetre), of the particle;
- g is the acceleration due to gravity, in metres per second squared;
- d_n is the limiting Stokes diameter, in micrometres, corresponding to the *n*th sample extraction.

9.5 Calculation of cumulative percentage undersize

The cumulative percentage by mass, p_n , of particles smaller than each of the limiting Stokes diameters d_n for each time interval t_n is given by the formula

$$\rho_n = \frac{m_n \times V_s}{m_s \times V_p} \times 100$$

where

- m_n is the mass, in grams, of the fraction corrected for the mass of the dispersing agent;
- $m_{\rm s}$ is the mass, in grams, of the test portion;
- $V_{\rm p}$ is the volume, in millilitres, of the pipette;
- $V_{\rm s}$ $\,$ is the volume, in millilitres, of the sedimentation vessel.

9.6 Repetition of test

Repeat the procedure on a further amount of suspension prepared from the same sample. The results of the test shall be accepted only if the two values for each of the percentages by mass, of particles smaller than the same limiting Stokes diameters, do not differ by more than 4 %.

9.7 Expression of results

Plot the results of the analysis with the micrometric sizes as abscissae and the percentages undersize as ordinates. From the smooth curve drawn through the points, select the cumulative percentages corresponding to the series required.

Report the results to the nearest 1 %.

10 Determination of matter volatile at 105 $^\circ \mathrm{C}$

See Part II of ISO 787. By agreement between the interested parties, amounts other than 10 g may be used for the test.

11 Determination of loss on ignition

11.1 Apparatus

11.1.1 Porcelain or platinum dish, shallow.11.1.2 Muffle furnace

11.2 Procedure

Weigh, to the nearest 0,001 g, about 2 g of the test sample, previously dried in accordance with clause 10, into the tared platinum or porcelain dish (11.1.1), ignite in the muffle furnace (11.1.2) at 1 000 \pm 25 °C to constant mass and cool in a desiccator containing phosphorus pentoxide²⁾.

NOTE In the case of extender No. 18, retain the ashed residue for the determination of silica (clause 17).

11.3 Expression of results

The loss on ignition, as a percentage by mass, is given by the formula

$$100 (m_0 - m_1)$$

m_o

where

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in grams, of the test portion after ignition.

12 Determination of matter soluble in water

12.1 General remarks

12.1.1 Methods to be used

The methods specified in **12.2**, **12.3** and **12.4** are intended to be used with the various extenders as indicated in Table 3.

12.1.2 *Water*

Freshly boiled distilled water or water otherwise prepared of equal purity shall be used.

NOTE Distilled water stored under ordinary laboratory conditions is likely to absorb carbon dioxide, ammonia, etc. It is recommended that the distilled water used for the test be boiled to expel dissolved gases and then cooled in a clean atmosphere. A soda-lime tube fitted to the flask is a satisfactory way of ensuring that no carbon dioxide is absorbed during cooling. As a further precaution, the pH value of the water itself should be checked before use. The water should have a pH value between 6,0 and 7,0.

Table 3 — Test method to be used for determination of matter soluble in water in each extender

		Test method					
	Extender	Hot	Cold extraction				
No.		Method 1 (see 12.2)	Method 1A (see 12.3)	method (see 12.4)			
1	Barytes	×	—	—			
2	Blanc fixe	×	In addition, if necessary, to Method 1	—			
3	Whiting	—	—	×			
4	Calcium carbonate, crystalline	—	—	×			
5	Calcium carbonate, precipitated	—	—	×			
6	Dolomite	—	—	×			
7	China clay	×	—	—			
8	Talc, fibrous form	×	—	—			
9	Talc, lamellar form	×	—	—			
10	Talc, containing carbonate	—	—	×			
11	Mica	×	—	—			
12	Silica (quartzite), type 1	×	—	—			
13	Silica (quartzite), type 2	×	—	—			
14	Kieselguhr, type 1	×	—	—			
15	Kieselguhr, type 2	×					
16	Calcium silicate, synthetic	No simula m	the daire a sticle of				
17	Sodium aluminium silicate, synthetic		ethod gives satisfactory res				
18	Silica, synthetic	extenders; therefore, no method is specified.					

 $^{^{2)}}$ When testing extenders containing calcium carbonate, care should be taken to avoid hydration of the quicklime formed.

12.2 Method 1 (hot extraction method)

See Part III of ISO 787.

The test portion shall consist of about 10 g, weighed to the nearest 0,01 g.

12.3 Method 1A (for determination of water-soluble alkaline earth metals in extender No. 2)

NOTE In the case of barium sulphate, precipitated (blanc fixe), if the matter soluble in water as calculated in **12.2** exceeds 0,5 %, an additional determination of water-soluble alkaline earth compounds is required, using the procedure specified below. The difference between the two results is then reported as the matter soluble in water in the material, as indicated in the notes to Table 1.

12.3.1 Reagents

12.3.1.1 Hydrochloric acid, 1 + 1

Add 1 volume of hydrochloric acid, ρ 1,18 g/ml, to 1 volume of water.

12.3.1.2 *Ammonia solution*, 1 + 1

Add 1 volume of ammonia solution, ρ 0,88 g/ml, to 1 volume of water.

12.3.1.3 Ammonium oxalate, 10 g/l solution

12.3.1.4 *Ammonium oxalate*, crystals.

12.3.2 Apparatus

12.3.2.1 Pipette, 50 ml.

12.3.2.2 Muffle furnace

12.3.2.3 Platinum crucible

12.3.3 Procedure

Place 50 ml of the diluted filtrate obtained from the procedure referred to in **12.2** into a 250 ml beaker and cover with a clock-glass. Add slowly 5 ml of the hydrochloric acid (**12.3.1.1**) and boil for a few minutes. Dilute with hot water to about 150 ml and bring nearly to the boil.

Add 2 g of ammonium oxalate crystals (**12.3.1.4**) and stir until dissolved. Add the ammonia solution (**12.3.1.2**) while stirring until the solution is alkaline to methyl red. Allow to stand firstly on a steam-bath or water-bath for at least 1 h, and then in the cold for at least 4 h. Filter through a fine-textured, ashless filter paper and wash the precipitate and filter paper with the ammonium oxalate solution (**12.3.1.3**). Ignite in the platinum crucible (**12.3.2.3**) in the muffle furnace (**12.3.2.2**) at a temperature of not less than 1 000 °C. Cool in a desiccator and weigh to the nearest milligram.

12.3.4 Expression of results

The content of water-soluble alkaline earth compounds, expressed as calcium sulphate $({\rm CaSO_4})$ as a percentage by mass, is given by the formula

$$\frac{12,14\ m_1 \times 100}{m_0} = 1\ 214\frac{m_1}{m_0}$$

where

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

 m_1 is the mass, in grams, of the precipitate after ignition.

12.4 Method 2 (Cold extraction method)

$12.4.1\,Apparatus$

12.4.1.1 One-mark volumetric flask, 500 ml, with stopper.

12.4.1.2 Flat-bottomed evaporating dish, of glass, glazed porcelain or silica.

12.4.2 Procedure

Weigh, to the nearest 0,01 g, about 10 g of the test sample into the 500 ml stoppered flask (**12.4.1.1**). Add 5 ml of ethanol, 95 % (V/V), to wet the sample thoroughly. Add 245 ml of water and stir for 1 h at room temperature by means of a magnetic stirrer.

Mix thoroughly by shaking and inversion and filter through a fine-textured filter paper, returning the filtrate to the filter paper until it runs clear.

Evaporate 100 ml of the perfectly clear filtrate to dryness in the tared, flat-bottomed dish (12.4.1.2), on a water-bath.

Dry the residue in an oven at 105 ± 2 °C, cool in a desiccator and weigh to the nearest 1 mg. Repeat the heating, cooling and weighing operations until the results of the two last weighings, at an interval including at least 30 min heating, do not differ by more than 10 % of the final figure obtained for the matter soluble in water.

12.4.3 Expression of results

The content of matter soluble in water, as a percentage by mass, is given by the formula

$$250 \ \frac{m_1}{m_0}$$

where

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

 m_1 is the mass, in grams, of the residue.

13 Determination of pH of aqueous suspension

See Part IX of ISO 787. It is recommended, however, that the suspension should be allowed to stand for 10 min instead of 5 min before making the determination.

14 Determination of barium sulphate in extenders Nos. 1 and 2

14.1 Introduction

For extenders Nos. 1 and 2, the acid-insoluble barium, calculated as barium sulphate, is accepted as the $BaSO_4$ content.

14.2 Reagents

14.2.1 Fusion mixture

Mix equal parts by mass of sodium carbonate and potassium carbonate.

14.2.2 Sodium carbonate, solution.

Dissolve 1 g of anhydrous sodium carbonate in 100 ml of water.

14.2.3 Hydrochloric acid, ρ 1,18 g/ml.

14.2.4 Hydrochloric acid, 1 + 3.

Dilute 1 volume of hydrochloric acid, ρ 1,18 g/ml, with 3 volumes of water.

14.2.5 *Hydrochloric acid*, 1 + 100.

Dilute 1 volume of hydrochloric acid, ρ 1,18g/ml, with 100 volumes of water.

14.2.6 Sulphuric acid, 1 + 9.

Add 1 volume of sulphuric acid, ρ 1,84 g/ml, slowly to 9 volumes of water.

14.2.7 *Ammonia solution*, 1 + 3.

Add 1 volume of ammonia solution, ρ 0,88 g/ml, to 3 volumes of water.

14.2.8 Bromine water, saturated solution.

14.2.9 Hydroxylammonium chloride (NH $_2$ OH·HCl), 50 g/l solution.

14.3 Apparatus

14.3.1 Platinum crucible, with lid

14.3.2 Sintered silica crucible, pore size index P 16 (diameter of pores 10 to $16 \,\mu$ m).

14.4 Procedure

14.4.1 Stage 1

14.4.1.1 *EXTENDER No. 1*

Into a 400 ml beaker, weigh, to the nearest 0,001 g, about 1 g of the test sample previously dried in accordance with clause 10. Add 100 ml of the dilute hydrochloric acid (**14.2.5**) and allow to stand for 1 h at room temperature, with occasional stirring. Filter through a 125 mm fine-textured ashless filter paper and wash once with 10 ml of hot water.

Transfer the filter paper to the platinum crucible (14.3.1), dry and ignite. Add 5 g of the fusion mixture (14.2.1), mix well, and then place a further 1 g of the fusion mixture on top of the mixture in the crucible.

Continue as given in Stage 2 below.

14.4.1.2 *EXTENDER No. 2*

Weigh 5 g of the fusion mixture (14.2.1) into the platinum crucible (14.3.1). Add approximately 1 g, weighed to the nearest 0,001 g, of the test sample, previously dried in accordance with clause 10. Mix well and place a further 1 g of the fusion mixture on top of the mixture in the crucible.

Continue as given in Stage 2 below.

14.4.2 Stage 2

14.4.2.1 EXTENDERS Nos. 1 AND 2

Heat the crucible, fitted with its lid, at bright red heat for 15 min. Then allow the crucible and lid to cool.

Measure 100 ml of hot water into a 600 ml beaker, A, and place into it the crucible and lid. Heat gently for 15 min to extract the melt, then, using clean nickel or other suitable tongs, remove the crucible and lid. Rinse the crucible and lid and tongs with water into the beaker. Remove any white residue with a rubber-tipped stirring rod and rinse again.

Measure 95 ml of water and 5 ml of the hydrochloric acid (14.2.3) into a second 600 ml beaker, B, and put the crucible and lid into it.

Allow the aqueous fusion extract in beaker A to settle on a warm surface for 30 min. Decant the hot solution through a 125 mm fine-textured ashless filter paper. Wash the precipitate three times by decantation, using 10 ml portions of the hot sodium carbonate solution (14.2.2) and transfer to the filter paper. Repeat the washing three times with 5 ml portions of the sodium carbonate solution and discard the washings.

Remove the crucible and lid from the acid solution in beaker B and wash down with water into the beaker. Transfer this solution to beaker A, in which the melt was extracted, and add to it the filter paper and residue. Cover with a clock-glass, boil for 5 min and then filter through a 125 mm filter paper into beaker B, which originally contained the acid solution. Wash the filter papers thoroughly six times with 10 ml portions of hot water.

Transfer the filter papers to the platinum crucible and dry, then ignite at dull red heat and add 2 g of the fusion mixture (14.2.1) to the crucible. Heat the crucible and lid at bright red heat for 15 min. Allow the crucible and lid to cool.

Measure 50 ml of hot water into beaker A and place into it the crucible and lid. Heat gently for 15 min to extract the melt. Filter through a 125 mm filter paper. Wash twice with 5 ml portions of the sodium carbonate solution (14.2.2).

Add to the beaker A (which still contains the crucible and lid), 20 ml of the dilute hydrochloric acid (14.2.4) and 50 ml of hot water. Pour this solution through the filter paper, and collect the filtrate in the beaker B which contained the original acid extract. Wash six times with 5 ml portions of hot water. Dilute the filtrate to approximately 300 ml.

14.4.2.2 EXTENDER No. 1 ONLY (this stage shall be omitted for extender No. 2)

Boil the solution in beaker B to remove carbon dioxide and then add bromine water (14.2.8) until a yellow colour appears. Add the ammonia solution (14.2.7) in slight excess and boil to precipitate the hydroxides of iron, aluminium and manganese. Filter the precipitate on a fast filter paper and wash three times with hot ammonia solution and collect the filtrate in a 400 ml beaker. Dissolve the precipitate into another beaker with approximately 8 ml of hydrochloric acid (14.2.4) and 12 ml of water to which a few drops of hydroxylammonium chloride solution (14.2.9) have been added. Dilute this solution to 50 ml, add bromine water until a yellow colour appears and then add the ammonia solution in slight excess. Filter and wash the precipitate as described above. Add the first and second filtrates to beaker B and acidify the whole with hydrochloric acid (14.2.4) and proximately 4 ml of acid in excess.

14.4.2.3 EXTENDERS Nos. 1 AND 2

Heat the acid extract to boiling. Stir and add slowly 20 ml of the hot sulphuric acid solution (14.2.6). Allow the covered beaker to stand on a warm surface for 3 h.

Filter through the tared sintered silica crucible (14.3.2). Wash four times with 10 ml portions of hot water. Dry in an oven at 105 ± 2 °C for 15 min and ignite in a muffle furnace at a temperature of 500 to 600 °C to constant mass. Cool in a desiccator and weigh. Alternatively, the precipitate may be filtered on a filter paper, placed in a platinum crucible, dried and ignited before cooling in a desiccator.

14.5 Expression of results

The content of barium compounds, expressed as barium sulphate $(BaSO_4)$, as a percentage by mass, is given by the formula

$$\frac{100\ m_1}{m_0}$$

where

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

 m_1 is the mass, in grams, of the precipitate.

15 Determination of calcium carbonate content of extenders Nos. 3 and 5

15.1 Reagents

15.1.1 Sodium carbonate, N solution.

NOTE Sodium hydroxide may be used in place of the sodium carbonate solution; it gives a more vivid end-point but needs standardizing more frequently.

15.1.2 Hydrochloric acid, approximately M solution.

15.1.3 *Mixed indicator,* prepared by mixing 1 part of dimethyl yellow, 2 g/l, in ethanol, 96 % (V/V), with 4 parts of bromocresol green, 2 g/l, in ethanol, 96 % (V/V).

15.2 Apparatus

15.2.1 Flask, 150 ml, flat-bottomed with a wide neck, fitted with a ground glass socket.

15.2.2 Condenser, glass, fitted with a ground glass cone, to fit the flask (15.2.1).

15.2.3 Pipette, 50 ml.

15.3 Procedure

Into the 150 ml flask (15.2.1), weigh, to the nearest 0,001 g, between 2,0 and 2,2 g of the test sample, previously dried in accordance with clause 10. Pipette 50 ml of the hydrochloric acid (15.1.2) onto the sample, and swirl gently after fitting the condenser (15.2.2) onto the flask. Warm gently and bring to the boil. Then simmer for 5 min and allow to cool. Finally, wash down the inside of the condenser with cold water, and disconnect the flask.

Add 3 drops of the mixed indicator (15.1.3) to the flask and titrate with the sodium carbonate solution (15.1.1), until the colour changes from red to yellowish-green (volume needed is V_2).

Titrate 50 ml of the hydrochloric acid solution with the sodium carbonate solution (volume needed is V_1).

 $NOTE \quad An automatic titration unit using a glass electrode and an end-point of pH 4,5 permits a method with greater accuracy and may be used instead of the above method.$

15.4 Expression of results

The content of carbonates, expressed as calcium carbonate ($CaCO_3$) as a percentage by mass, is given by the formula

$\frac{5,004 (V_1 - V_2) T}{m}$

where

 V_1 is the volume, in millilitres, of the sodium carbonate solution (15.1.1) required to neutralize 50 ml of the acid;

 V_2 is the volume, in millilitres, of the sodium carbonate solution (15.1.1) required to neutralize the excess of acid after the addition of the sample;

T is the exact normality of the sodium carbonate solution (15.1.1);

m is the mass, in grams, of the test portion.

16 Determination of calcium content of extender No. 4 and calcium and magnesium contents of extenders Nos. 6 and 10

16.1 Reagents

16.1.1 Ammonium chloride

16.1.2 Sodium chloride

 $\label{eq:16.1.3} \textit{EDTA} (ethylenediamine-\textit{NNN'N'}-tetra-acetic acid, disodium salt), 0,05 \ M \ solution \ standardized against a standard magnesium solution using the procedure specified in 16.2.3.$

16.1.4 Ammonia solution, ρ 0,880 g/ml.

16.1.5 Hydrochloric acid, 6 M solution.

16.1.6 Potassium hydroxide, 4 M solution.

16.1.7 $Calcon^{3)}$ (or other appropriate indicator).

Triturate 1 part of Calcon with 99 parts of anhydrous sodium sulphate.

16.1.8 *Mordant black II* (Erichrome Black T)⁴⁾ (or other appropriate indicator).

Triturate 1 part of Mordant black II with 99 parts of sodium chloride to a fine powder.

16.1.9 *Indicator paper*, pH 9,5 to 13.

16.2 Procedure

16.2.1 Preparation of filtrate

Into a beaker, weigh, to the nearest 0,1 mg, about 0,5 g of the test sample, previously dried in accordance with clause **10**. Moisten it with a little water, add 7 ml of the hydrochloric acid (**16.1.5**), cover the beaker with a watch-glass and boil for a few minutes. Dilute to 100 ml.

Add 3 g of the ammonium chloride (16.1.1) and neutralize with ammonia solution (16.1.4). Then add 1 ml excess of the ammonia solution to precipitate iron and aluminium. Boil, filter rapidly, and wash the precipitate with hot water. Collect the filtrate and washings, make slightly acid, and dilute to 500 ml in a one-mark volumetric flask.

16.2.2 Determination of calcium carbonate (for extender No. 4) or calcium compounds as calcium carbonate in extenders Nos. 6 and 10

16.2.2.1 TITRATION

By means of a pipette, transfer 100 ml of the filtrate obtained in **16.2.1** to a 400 ml beaker. Dilute to about 250 ml, and add approximately 10 ml of the potassium hydroxide solution (**16.1.6**) in order to obtain a minimum pH value of 12,0. Add 0,2 to 0,4 g of the Calcon mixture (**16.1.7**) (see note).

Titrate with the EDTA solution (16.1.3) with constant stirring until the colour changes from wine-red to clear blue (volume required is V_1).

 $^{^{3)}}$ Sodium 1-(2-hydroxy-1-naphthylazo)-2-naphthyl-4-sulphonate.

⁴⁾ Sodium 1-(1-hydroxy-2-naphthylazo)-6-nitronaphthalene-4-sulphonate.

NOTE Add a small amount of a soluble magnesium salt to intensify the colour if the calcium carbonate content of pure calcite is being determined.

16.2.2.2 EXPRESSION OF RESULTS

The content of calcium carbonate or the content of carbonates, expressed as $CaCO_3$ as a percentage by mass, is given by the formula

50,04 V₁ T

where

- V_1 is the volume, in millilitres, of EDTA solution (16.1.3) added;
- *m* is the mass, in grams, of the test portion;
- T is the exact molarity of the EDTA solution (16.1.3).

16.2.3 Determination of magnesium compounds in extenders Nos. 6 and 10

NOTE By titration of the filtrate against the same EDTA solution as used in **16.2.2.1** for calcium compounds but using a different indicator and a different pH value, the magnesium and calcium contents may be determined together. The magnesium content is then determined by subtraction of the calcium content.

16.2.3.1 TITRATION

By means of a pipette, transfer 100 ml of the filtrate obtained in **16.2.1** into a 400 ml beaker and dilute with water to about 250 ml. Add ammonia solution (**16.1.4**) to adjust the pH to between 10 and 11 (up to 20 ml may be required).

Titrate with the EDTA solution (16.1.3). Add the Mordant black II (16.1.8) indicator towards the end of the titration. The end-point is reached when the colour changes from wine-red to blue (volume required is V_2).

NOTE The change in colour is not so marked as for the titration of calcium carbonate using Calcon as indicator.

16.2.3.2 EXPRESSION OF RESULTS

The content of magnesium compounds, expressed as magnesium carbonate ($MgCO_3$) as a percentage by mass, is given by the formula

 $\frac{42,16(V_2 - V_1)T}{m}$

where

- m is the mass, in grams, of the test portion;
- V_1 is the volume, in millilitres, of EDTA solution (16.1.3) added in 16.2.2.1;
- V_2 is the volume, in millilitres, of EDTA solution (16.1.3) added in 16.2.3.1;

T is the exact molarity of the EDTA solution (16.1.3).

17 Determination of silica in extender No. 18

17.1 Reagents

17.1.1 *Sulphuric acid,* 1 + 1.

Add 1 volume of sulphuric acid, ρ 1,84 g/ml, slowly to 1 volume of water.

17.1.2 *Hydrofluoric acid*, 40 % (*m/m*).

17.2 Apparatus

17.2.1 Platinum dish

17.2.2 Muffle furnace

17.3 Procedure

Transfer the residue obtained in clause 11 (determination of loss on ignition) to the platinum dish (17.2.1) and add about 1 ml of the sulphuric acid (17.1.1). Heat the dish gently until fuming ceases and then continue the heating at 900 °C for 15 min in the muffle furnace (17.2.2). Remove from the furnace, cool in a desiccator and weigh (m_2) .

Add to the residue in the platinum dish 15 ml of the hydrofluoric acid (**17.1.2**) and 1 ml of the sulphuric acid (**17.1.1**) and evaporate to a syrup, taking care to avoid loss by spitting. Cool the dish and wash the sides down with water. Then add a further 10 ml of the hydrofluoric acid and evaporate to dryness. Heat the residue on a hot-plate until white fumes are no longer evolved, then ignite in the muffle furnace at 900 °C for 15 min.

Remove from the furnace, cool in a desiccator and weigh (m_3) .

17.4 Expression of results

The silica content, as a percentage by mass, is given by the formula

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

where

 m_1 is the mass, in grams, of ignited residue obtained in **11.2**;

 m_2 is the mass, in grams, after igniting with sulphuric acid;

 m_3 is the mass, in grams, after igniting with hydrofluoric acid.

Annex Oil absorption values

The normal ranges of oil-absorption when determined by the method specified in Part V of ISO 787 are given below.

No.	Extender	Oil absorption grams of oil per 100 g
1	Barytes	8 to 15
2	Blanc fixe	15 to 30
3	Whiting	12 to 22
4	Calcium carbonate, crystalline	10 to 22
5	Calcium carbonate, precipitated	30 to 80
6	Dolomite	10 to 22
7	China clay	30 to 60
8	Talc, fibrous form	37 to 42
9	Talc, lamellar form	
	grade a grade b grade c	35 to 43 35 to 72 35 to 72
10	Talc, containing carbonate	39 to 42
11	Mica, wet ground, grade a Mica, dry ground, grade b	55 to 75 45 to 90
12 and 13	Silica (quartzite), types 1 and 2	14 to 25
14 and 15	Kieselguhr, types 1 and 2	60 to 100
16	Calcium silicate, synthetic	70 to 250
17	Sodium aluminium silicate, synthetic	100 to 250
18	Silica, synthetic	100 to 250

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