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Extenders for paints

Matières de charge pour peintures

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

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Extenders for paints

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, $3MgO{\cdot}4SiO_2{\cdot}H_2O$.

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

Magnesium silicate, natural, hydrated, $3MgO{\cdot}4SiO_2{\cdot}H_2O$.

Extender No. 10 : Talc, containing carbonate

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

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

Potassium aluminium silicate, natural, hydrated, $K_2O{\cdot}3Al_2O_3{\cdot}6SiO_2.2H_2O$, 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, SiO_2 content equal to or greater than 98 %.

Extender No. 14 : Kieselguhr, type 1 (grades a, b, c, d)

Diatomaceous siliceous earth. 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.

TABLE 1 – **Composition and properties of extenders**

No.	Extender	Composition % (*m*/*m*)	Test method	Colour (see clause 7)	Residue on sieve[1] (see clause 8) % (*m*/*m*) max. 125 µm	Residue on sieve 63 µm	Residue on sieve 45 µm	Particle size distribution[2] (see clause 9) % (*m*/*m*) min. < 20 µm	< 10 µm	< 5 µm	< 2 µm[3]	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)
1	Barytes	$BaSO_4$, 90 min.	see clause 14	No extender to be darker or more strongly coloured than an agreed sample.											
	grade a				–	–	2,0	65	40	20	(5)	0,5	0,5 max.	0,5	6 to 10
	grade b				–	–	0,5	99	95	80	(20)				
2	Blanc fixe	$BaSO_4$, 95 min.	see clause 14		0,02	0,1	0,2	90	80	60	(25)	0,5	–	0,5[4]	5,5 to 9,5
3	Whiting	$CaCO_3$, 96 min. 99 max.	see clause 15												
	grade a				0,5	6	15	–	–	–	–	0,3	42 to 44	0,15	8,0 to 9,5
	grade b				0,01	0,5	2	85	65	45	(20)				
	grade c				–	0,05	0,5	90	80	55	(25)				
	grade d				–	–	0,01	97	95	70	(35)				
4	Calcium carbonate (crystalline)	$CaCO_3$, 98 min.	see clause 16												
	grade a				0,01	1,0	5,0	70	40	25	(10)	0,3	42 to 44,5	0,15	8 to 10
	grade b				0,01	0,5	1,5	80	45	30	(10)				
	grade c				–	0,01	0,1	95	65	35	(15)				
	grade d				–	–	0,01	–	98	70	(25)				
5	Calcium carbonate (precipitated)	$CaCO_3$, 97 min. 100 max.	see clause 15												
	grade a				0,1	0,25	0,5	90	70	40	–	1,0	43,5 to 44,5	0,3	8 to 10,5
	grade b				0,1	0,25	0,5	–	90	70	(20)				
6	Dolomite	$CaMg(CO_3)_2$, 97 min.[5]	see clause 16												
	grade a				0,01	1,0	5,0	70	40	25	(10)	0,3	46 to 48	0,2	8 to 10,5
	grade b				0,01	0,5	1,5	80	45	30	(10)				
	grade c				–	0,01	0,1	95	65	35	(15)				
	grade d				–	–	0,01	–	98	70	(25)				
7	China clay														
	grade a				–	–	0,5	90	70	–	(15)	2	10 to 14	0,5	4,5 to 9,5
	grade b				–	–	0,1	95	80	–	(35)				
	grade c				–	–	0,05	99,5	99	–	(70)				
8	Talc, fibrous form														
	grade a				–	–	1,5	75	55	40	(20)	0,5	4 to 7	0,5	8,5 to 10
	grade b				–	–	0,5	90	75	50	(25)				
9	Talc, lamellar form														
	grade a				0,01	0,5	3,0	80	50	30	(8)	0,5	4 to 8	0,5	8,5 to 10
	grade b				–	0,01	0,10	95	70	40	(15)				
	grade c				–	–	0,01	–	95	70	(25)				
10	Talc, containing carbonate	Magnesium silicate with a max. of 50 % $MgCO_3 + CaCO_3$	see clause 16												
	grade a				0,01	0,5	3,0	80	50	30	(8)	0,5	28 max.	0,5	8 to 10,5
	grade b				–	0,01	0,10	95	70	40	(15)				
	grade c				–	–	0,01	–	95	70	(25)				

TABLE 1 *(continued)*

No.	Extender	Composition % (*m*/*m*)	Test method	Colour (see clause 7)	Residue on sieve[1] (see clause 8) % (*m*/*m*) max.			Particle size distribution[2] (see clause 9) % (*m*/*m*) min.				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)
					125 µm	63 µm	45 µm	< 20 µm	< 10 µm	< 5 µm	< 2 µm[3]				
11	Mica		Any recognized classical method	No extender to be darker or more strongly coloured than an agreed sample.											
	grade a				–	1,0	3,0	98	80	30	(5)	0,5	5,5 max.	0,5	7 to 9,5
	grade b				–	–	0,5	98	70	30	(10)				
12	Silica (quartzite) type 1	SiO$_2$, 80 to 98													
	grade a				40	–	–	–	–	–	–	0,5	5 max.	0,5	7 to 10
	grade b				2	25	60	–	–	–	–				
	grade c				–	0,5	5,0	70	–	10	–				
	grade d				–	–	0,1	–	–	95	–				
13	Silica (quartzite) type 2	SiO$_2$, 98 min.													
	grade a				40	–	–	–	–	–	–	0,5	0,2 max.	0,5	7 to 8,5
	grade b				2	25	60	–	–	–	–				
	grade c				–	0,5	5,0	70	–	10	–				
	grade d				–	–	0,1	–	–	95	–				
14	Kieselguhr, type 1	SiO$_2$, 70 to 80													
	grade a				–	–	15	70	10	–	–	12	15 max.	1,0	6,5 to 10
	grade b				–	–	5	75	20	5	–				
	grade c				–	–	1	80	30	10	–				
	grade d				–	–	1	90	50	30	–				
15	Kieselguhr, type 2	SiO$_2$, 80 min.													
	grade a				–	–	15	70	10	–	–	2	1 max.	1,0	6,5 to 10
	grade b				–	–	5	75	20	5	–				
	grade c				–	–	1	80	30	10	–				
	grade d				–	–	1	90	50	30	–				
16	Calcium silicate, synthetic	To be agreed between the interested parties			–	1,0	3,0	90	70	40	–	12	8 max.	To be agreed between the interested parties	8 to 12,5
17	Sodium aluminium silicate, synthetic				–	2,0	4,0	85	50	20	–	12	8 max.		8 to 12,5
18	Silica, synthetic	SiO$_2$, 97 min.	see clause 17		–	2,0	5,0	80	50	20	–	12	8 max.		3,5 to 8,0

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1) 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. Part 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.

2) The method for the determination of particle size distribution is based on Stokes' law. Strictly speaking, Stokes' 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.

3) The 2 µm limits are given for guidance only.

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

5) The proportion by mass of CaCO$_3$ to MgCO$_3$ is in practice between 1,18 and 1,23 CaCO$_3$: 1,0 MgCO$_3$.

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 regents 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

8.1.1 Three sieves, 100 to 200 mm diameter, with sides of height 50 mm and meshes of apertures 45, 63 and 125 µm 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 µm).

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.

Dimensions in millimetres

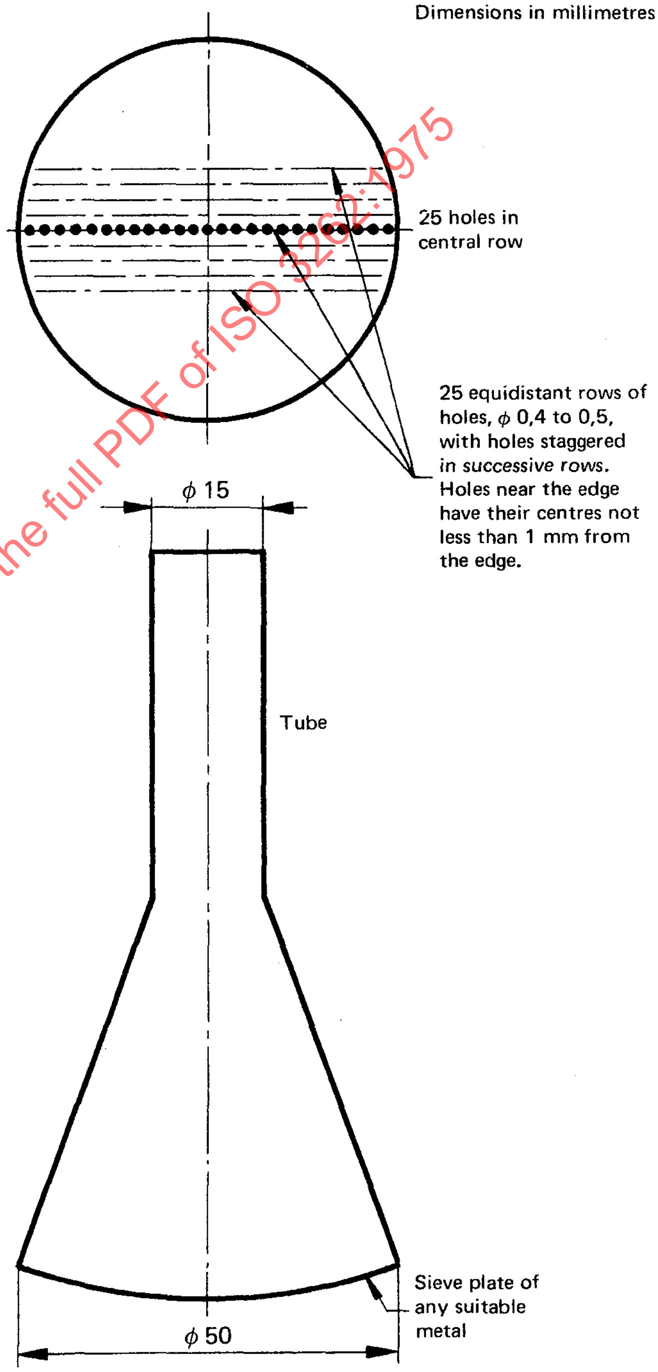

FIGURE 1 – **Rose used for washing the residue on sieve**

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[1] See ISO 48, *Vulcanized rubbers – Determination of hardness.*

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.

NOTES

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.

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 :

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

where

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

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

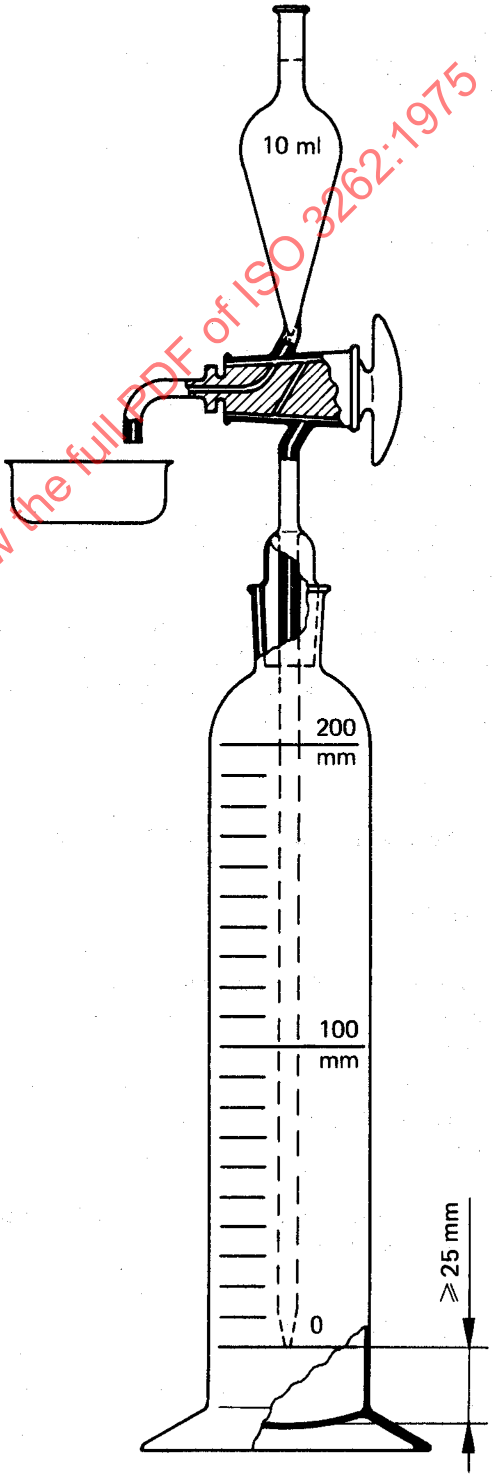

FIGURE 2 — **Sedimentation vessel and pipette**

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 up to 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 ± 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_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_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.

TABLE 2 – **Details for preparing dispersions for the determination of particle size**

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

Extender		**Mass to be used** (previously dried in accordance with clause 10) and weighed to an accuracy of 0,01 g g	**Mass and type of dispersing agent** (solid active material)
No.			
1	Barytes	20	0,75 g of sodium hexametaphosphate dissolved in 750 ml of water
2	Blanc fixe	20	
3	Whiting	3,5	0,07 to 0,14 g of suitable agent1) dissolved in 750 ml of water
4	Calcium carbonate, crystalline	3,5	
5	Calcium carbonate, precipitated	3,5	
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 dissolved in 750 ml of 20 % (*m*/*m*) ethanol in water2)
9	Talc, lamellar form	10	
10	Talc, containing carbonate	10	
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	5	0,75 g of sodium hexametaphosphate dissolved in 750 ml of water
14 and 15	Kieselguhr, type 1 and type 2		
16	Calcium silicate, synthetic		
17	Sodium aluminium silicate, synthetic		
18	Silica, synthetic		

NOTES

1) Certain sodium salts of polymethacrylates have been found most suitable. Such materials are sold as Polysaltz, Dispex, etc.

2) 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 withdrawal time*

The withdrawal time, t_n, in seconds, for the nth sample is given by the equation

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

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 nth sample extraction is made;

ρ_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 nth 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

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

where

m_n is the mass, in grams, of the fraction corrected for the mass of the dispersing agent;

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

V_p is the volume, in millilitres, of the pipette;

V_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 °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 ± 25 °C to constant mass and cool in a desiccator containing phosphorus pentoxide1).

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

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1) When testing extenders containing calcium carbonate, care should be taken to avoid hydration of the quicklime formed.

11.3 Expression of results

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

$$\frac{100\,(m_0 - m_1)}{m_0}$$

where

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

m_1 is the mass, in grams, of the 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**

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

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 ($CaSO_4$) 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 1,18 g/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 ($NH_2OH{\cdot}HCl$), 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 μm).

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.