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# INTERNATIONAL STANDARD



# 3262

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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.

International Standard ISO 3262 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and circulated to the Member Bodies in November 1973.

It has been approved by the Member Bodies of the following countries :

Austria	Italy	Spain
Brazil	Netherlands	Sweden
Bulgaria	New Zealand	Switzerland
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Ireland	Romania	U.S.S.R.
Israel	South Africa, Rep. of	Yugoslavia

The Member Body of the following country expressed disapproval of the document on technical grounds :

France

# 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  $\text{CaCO}_3$  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,  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , of lamellar crystalline form.

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

Magnesium silicate, natural, hydrated,  $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ .

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

Magnesium silicate, natural, hydrated,  $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ .

### Extender No. 10 : Talc, containing carbonate

Magnesium silicate, natural, hydrated,  $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ , lamellar form. Different from extender No. 9 in that it contains carbonate.

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

Potassium aluminium silicate, natural, hydrated,  $\text{K}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , 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.  $\text{SiO}_2$  content not less than 80 % but less than 98 %.

### Extender No. 13 : Silica (quartzite), type 2 (grades a, b, c, d)

Silica, natural,  $\text{SiO}_2$  content equal to or greater than 98 %.

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

Diatomaceous siliceous earth.  $\text{SiO}_2$  content not less than 70 % but less than 80 %.

### Extender No. 15 : Kieselguhr, type 2 (grades a, b, c, d)

Diatomaceous siliceous earth.  $\text{SiO}_2$  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 <sup>1)</sup> (see clause 8) (% (m/m) max.)				Particle size distribution <sup>2)</sup> (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				
1	Barytes grade a grade b	BaSO <sub>4</sub> , 90 min.	see clause 14	No extender to be darker or more strongly coloured than an agreed sample.	—	—	2,0	65	40	20	(5)	0,5	0,5 max.	0,5	6 to 10
2	Blanc fixe	BaSO <sub>4</sub> , 95 min.	see clause 14		0,02	0,1	0,2	90	80	60	(25)	0,5	—	0,54	5,5 to 9,5
3	Whiting grade a grade b grade c grade d	CaCO <sub>3</sub> , 96 min. 99 max.	see clause 15		0,5 0,01 — —	6 0,5 0,05 —	15 2 0,5 0,01	— 85 90 97	— 65 80 95	— 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 grade c grade d	CaCO <sub>3</sub> , 98 min.	see clause 16		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	42 to 44,5	0,15	8 to 10
5	Calcium carbonate (precipitated) grade a grade b	CaCO <sub>3</sub> , 97 min. 100 max.	see clause 15		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 grade a grade b grade c grade d	CaMg(CO <sub>3</sub> ) <sub>2</sub> , 97 min. <sup>5)</sup>	see clause 16		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	China clay grade a grade b grade c				— — —	— — —	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				— —	— —	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 grade a grade b grade c				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 <sub>3</sub> + CaCO <sub>3</sub>	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.	0,5	8 to 10,5

TABLE 1 (continued)

No.	Extender	Composition % (m/m)	Test method	Colour (see clause 7)	Residue on sieve <sup>1)</sup> (see clause 8) % (m/m) max.				Particle size distribution <sup>2)</sup> (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 <sup>3)</sup>				
11	Mica grade a grade b			No extender to be darker or more strongly coloured than an agreed sample.	—	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
12	Silica (quartzite) type 1 grade a grade b grade c grade d	SiO <sub>2</sub> , 80 to 98			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 <sub>2</sub> , 98 min.			40 2 — —	— 25 0,5 —	— 60 5,0 0,1	— — 70 —	— — — —	— — 10 95	— — — —	0,5	0,2 max.	0,5	7 to 8,5
14	Kieselguhr, type 1 grade a grade b grade c grade d	SiO <sub>2</sub> , 70 to 80			— — — —	— — — —	15 5 1 1	70 75 80 90	10 20 30 50	— 5 10 30	— — — —	12	15 max.	1,0	6,5 to 10
15	Kieselguhr, type 2 grade a grade b grade c grade d	SiO <sub>2</sub> , 80 min.			— — — —	— — — —	15 5 1 1	70 75 80 90	10 20 30 50	— 5 10 30	— — — —	2	1 max.	1,0	6,5 to 10
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 <sub>2</sub> , 97 min.	see clause 17		—	2,0	5,0	80	50	20	—	12	8 max.		3,5 to 8,0

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<sub>3</sub> to MgCO<sub>3</sub> is in practice between 1,18 and 1,23 CaCO<sub>3</sub> : 1,0 MgCO<sub>3</sub>.

## 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.

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## 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

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

**8.1.2 Glass rod**, tipped with rubber having a hardness of 50 IRHD<sup>1)</sup>.

**8.1.3 Filter crucible of sintered glass**, pore size index P 40 (diameter of pores 16 to 40  $\mu\text{m}$ ).

**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.

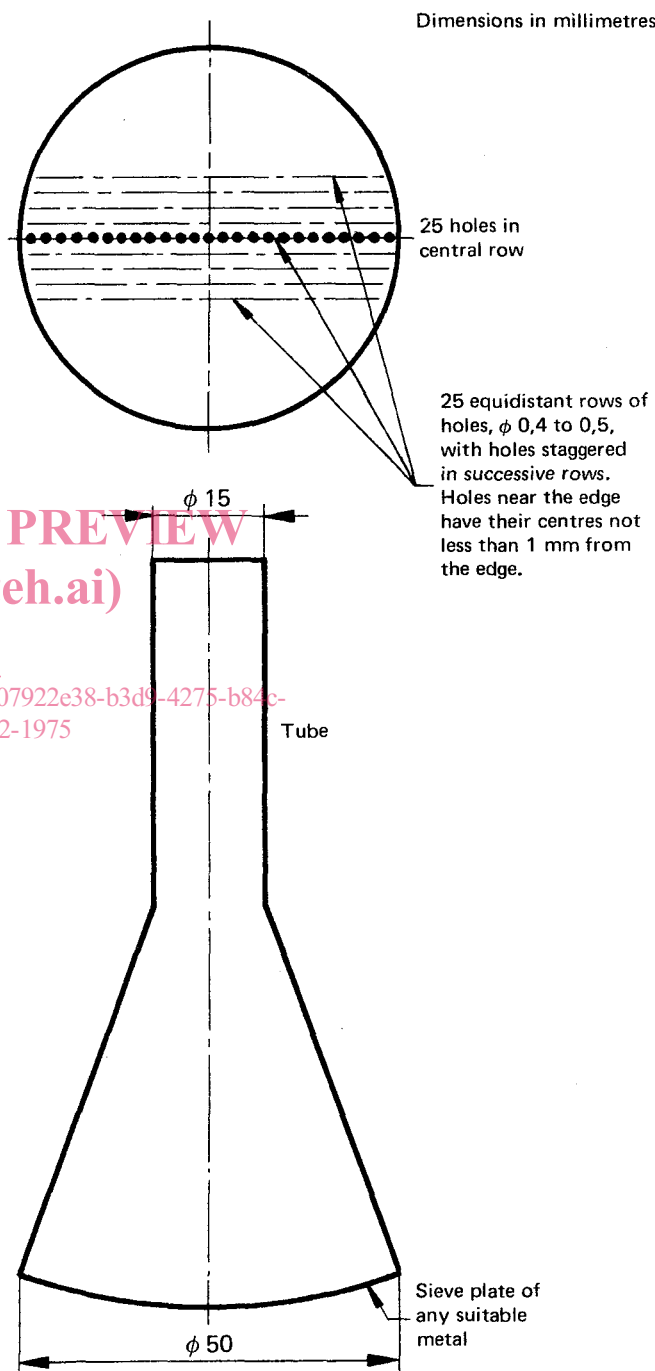


FIGURE 1 — Rose used for washing the residue on sieve

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  $105 \pm 2\text{ }^{\circ}\text{C}$ . 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).

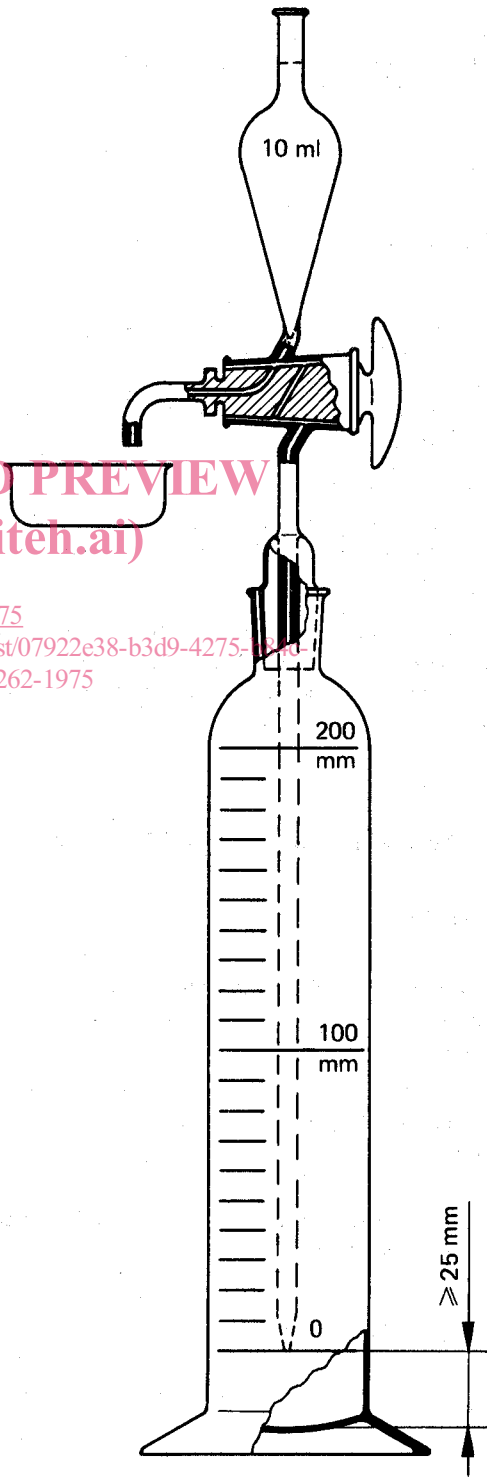


FIGURE 2 — Sedimentation vessel and pipette



**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  $23 \pm 0,5^{\circ}\text{C}$ , 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 ( $1\ 000 \pm 100$  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  $30^{\circ}$  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  $105 \pm 2^{\circ}\text{C}$  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  $23 \pm 0,5^{\circ}\text{C}$  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  $23 \pm 0,5^{\circ}\text{C}$  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  $20\ \mu\text{m}$  (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.