

INTERNATIONAL STANDARD

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Plastics — Polymer dispersions — Determination of sieve residue (gross particle and coagulum content)

*Plastiques — Dispersions de polymères — Détermination du résidu par
tamisage (teneur en grains et en coagulum)*



Reference number
ISO 4576:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4576 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 4576:1978), which has been technically revised.

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Plastics — Polymer dispersions — Determination of sieve residue (gross particle and coagulum content)

1 Scope

This International Standard specifies a method of determining the sieve residue (gross particle and coagulum content) of polymer dispersions, i.e. of particles much greater in diameter (for example 10 or 100 times) than the mean diameter of the other particles.

This determination is carried out by sieve analysis. The sieves used depend on the type of dispersion under test and are in consequence to be specified, or agreed upon by the interested parties, for each dispersion or group of closely related dispersions.

This International Standard refers only to coagulum-type content. Skinned material and fragments of skin larger than 5 mm are avoided while taking the sample (see clause 6)

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling*.

ISO 842:1984, *Raw materials for paints and varnishes — Sampling*.

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

3 Principle

The polymer dispersion is diluted with a specified volume of deionized water and filtered through a metal gauze of a specified mesh size. The residue is washed with deionized water and then dried and weighed.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and deionized water or water of equivalent purity.

4.1 Surfactants, for stabilizing the polymer dispersion during testing (if necessary), as agreed between the interested parties.

5 Apparatus

5.1 Test sieves (series of metal gauzes, preferably stainless steel), as specified in ISO 3310-1, in the form of discs or squares, including at least meshes with the following aperture sizes:

45 μm , 63 μm , 90 μm , 125 μm and 180 μm

Non-metallic gauzes may be used, but this shall be indicated in the test report.

5.2 Equipment to support the wire gauzes, such as steel rings or glass funnels of equal internal diameter between 25 mm and 50 mm.

5.3 Laboratory balances, one having an accuracy of 1 mg for weighing test portions of up to 200 g (or 10 mg for test portions between 200 g and 1 000 g) and the other having an accuracy of 0,1 mg for weighing the sieve (with and without the residue).

5.4 Normal oven or vacuum oven, capable of maintaining, to $\pm 2^\circ\text{C}$, a temperature of preferably 105°C , or any of the following alternative temperatures if more suitable for a particular dispersion:

80 $^\circ\text{C}$, 125 $^\circ\text{C}$ or 140 $^\circ\text{C}$

The temperature for drying the sieve residue shall be selected from those given, the preferred temperature being $105^\circ\text{C} \pm 2^\circ\text{C}$. The temperature chosen will depend on the stability of the polymer and the presence of any additives, and shall be in line with the temperature recommended for the determination of the residue on drying of the polymer dispersion under test.

5.5 Desiccator, big enough to hold the gauzes.

5.6 Beaker, of capacity at least 600 ml, with a lip.

5.7 Filtration apparatus, as shown in figure 1, for instance.

6 Sampling

Sampling shall be carried out in accordance with one of the methods specified in ISO 123 or ISO 842 to obtain a homogeneous sample. Visible skin and skin fragments larger than 5 mm shall be carefully avoided while sampling.

7 Procedure

7.1 Weigh approximately 100 g to 200 g (mass m_0) of the sample into the beaker (5.6).

NOTE 1 Depending on the required precision, larger test portions (up to 1 000 g) may be used.

7.2 Dilute the test portion with a volume, in millilitres, of water numerically equal to twice the mass of the test portion taken, adding a suitable surfactant (4.1) if necessary to the water before dilution.

NOTE 2 Dilution with water prevents film formation and promotes mobility, thus ensuring rapid and complete filtration. The surfactant prevents additional agglomeration or coagulation occurring during the test.

7.3 Carefully mix the added water (plus surfactant, if used) into the polymer dispersion, using a glass rod or a slowly rotating stirrer to produce adequate mixing without destroying existing agglomerates.

7.4 For each mesh size used, prepare a suitably sized, clean metal gauze test sieve (5.1), dry it in the oven (5.4) at $105^\circ\text{C} \pm 2^\circ\text{C}$ or any of the other specified temperatures to constant mass and, after cooling in the desiccator (5.5), weigh the test sieve to the nearest 0,1 mg (mass m_1).

If the metal gauze is not clean, immerse it for 30 min in boiling water and rinse it with acetone before drying to constant mass and weighing. For non-metallic gauzes, use appropriate cleaning procedures.

7.5 Place the sieve on a suitable support (5.2), and wet or wash the sieve with water or the appropriate surfactant solution (4.1).

Pour the diluted dispersion on to the sieve, ensuring that the liquid is poured through the centre of the sieve.

7.6 On completion of the filtration, wash the residue on the sieve with the same diluent as used for the test portion, followed by deionized water, until a clear filtrate is obtained.

7.7 Dry the sieve plus residue in the oven at $105^\circ\text{C} \pm 2^\circ\text{C}$, or at the alternative temperature selected, for approximately 30 min, weigh, and repeat the drying for 15 min periods until constant mass is reached.

7.8 Cool the sieve plus residue in the desiccator and weigh it to the nearest 0,1 mg (mass m_2).

7.9 Do not rinse the sieve without proper cleaning.

7.10 Carry out two determinations.

8 Expression of results

8.1 Calculate the sieve residue (gross particle and coagulum content) for a particular test sieve, expressed as a percentage by mass of the dispersion, using the formula

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (see 7.1);

m_1 is the mass, in grams, of the test sieve (see 7.4);

m_2 is the mass, in grams, of the sieve plus dried residue (coagulum) (see 7.8).

Dimensions in millimetres

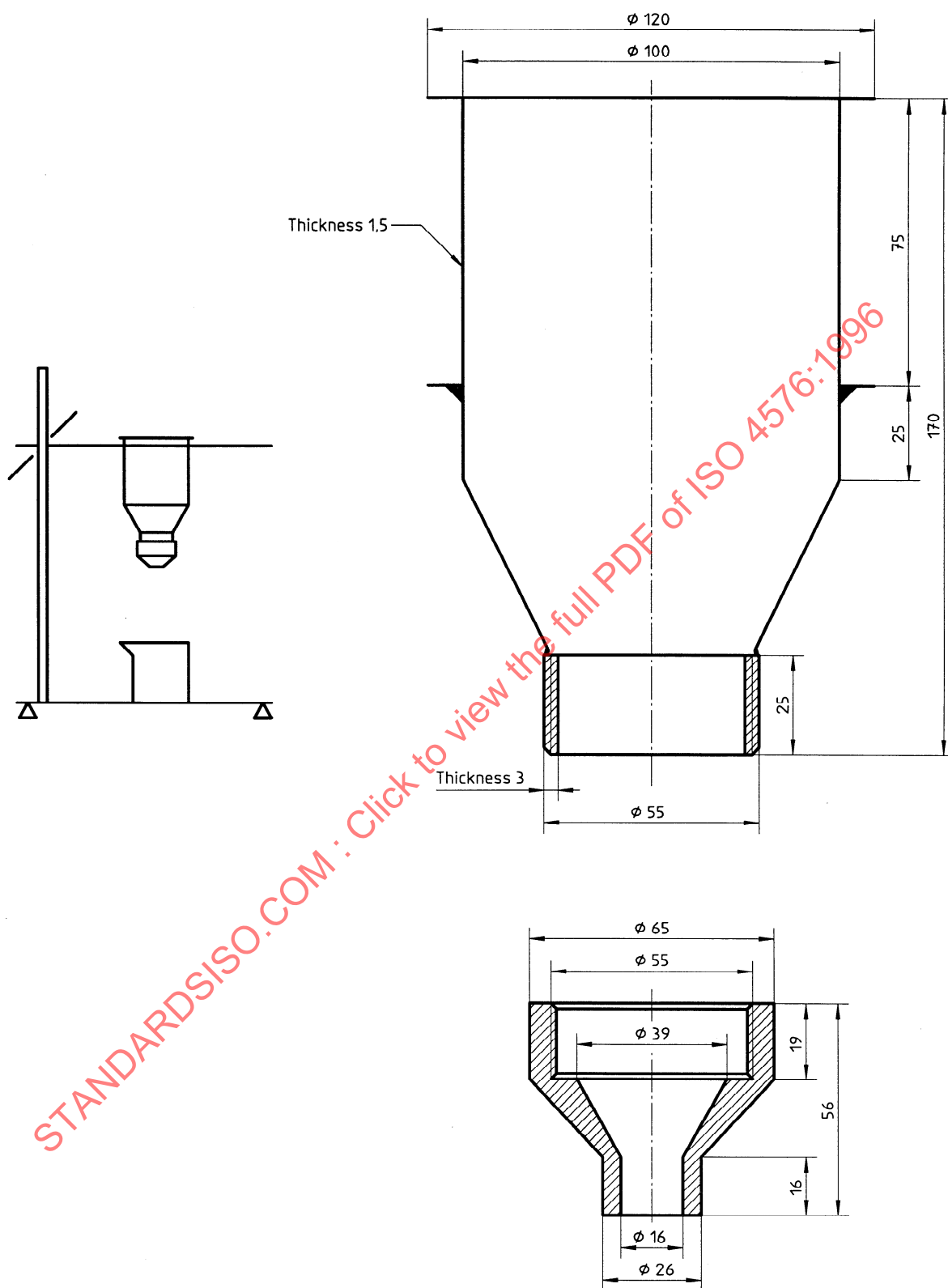


Figure 1 — Example of filtration apparatus

8.2 Calculate the arithmetic mean of the two determinations for each test sieve used, rounding to one place of decimals. The results of the individual determinations shall agree to $\pm 5\%$ of the mean value. If they do not, carry out two further determinations until the results satisfy this condition.

9 Precision

It is estimated that, if this test procedure is performed twice on the same day by the same person, using the same apparatus and testing the same laboratory sample, the two results will agree to within $\pm 8\%$.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for the identification of the polymer dispersion tested;
- c) the test sieve and drying temperature used;
- d) the sieve residue (gross particle and coagulum content);
- e) a description of the residue;
- f) any unusual feature noted during the determination;
- g) details of any deviation from the procedure specified in this International Standard or in the International Standards to which reference is made, as well as any operation considered as optional;
- h) the date and place of the test.

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