INTERNATIONAL STANDARD

ISO 14680-1

First edition
2000-04-01

Paints and varnishes — Determination of pigment content —

Part 1: Centrifuge method

Peintures et vernis — Détermination de la teneur en pigment —

Partie 1: Méthode par centrifugation

Reference number
ISO 14680-1:2000(E)

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Printed in Switzerland

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

Attention is drawn to the possibility that some of the elements of this part of ISO 14680 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14680-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 14680 consists of the following parts, under the general title *Paints and varnishes — Determination of pigment content*:

— *Part 1: Centrifuge method*

— *Part 2: Ashing method*

— *Part 3: Filtration method*

Paints and varnishes — Determination of pigment content —

Part 1:
Centrifuge method

1 Scope

This part of ISO 14680 is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a method for determining the pigment content of paints in which the solids are sedimented by centrifugation.

It is primarily intended for checking the composition during the production of coating materials and as an acceptance check for the coating material user. Difficulties may be encountered when the method is applied to coating materials containing dyestuffs, carbon black, very finely divided silicon dioxide or very finely divided titanium dioxide. It may not be suitable for emulsion paints.

The pigment content of coating materials can also be determined by an ashing method (see ISO 14680-2) or by a filtration method (see ISO 14680-3).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14680. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14680 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing*.

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

3 Term and definition

For the purposes of this part of ISO 14680, the following term and definition apply.

3.1
pigment content, determined by the centrifuge method
the proportion by mass of solid particles in the product under test which is insoluble in the solvent used for separation under the specified conditions

NOTE It includes pigments, extenders and other solid constituents of the product.

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

4 Principle

After diluting a test portion of the product under test with solvent, the solid particles are separated off by centrifuge, dried and weighed. The pigment content is then calculated from the mass of the solids component and that of the test portion.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Laboratory centrifuge, with an operating speed sufficient to ensure that the pigment separates from the binder and a relative centrifugal acceleration of about 250 000 (equivalent to 25 000 g). It shall be fitted with balanced swivelling buckets which can accommodate suitable tubes of e.g. 50 ml capacity.

5.2 Centrifuge tubes, with a capacity of e.g. 50 ml. These may be made of stainless steel, thick-walled glass, PTFE or other suitable inert material.

5.3 Polished-ball stirrer, consisting of a shaft of length about 150 mm and diameter about 5 mm having, on one end, a ball of diameter about 12 mm. The stirrer shall be made of a suitable material such as stainless steel or glass.

5.4 Drying oven, with forced ventilation, capable of being maintained at (125 ± 2) °C. The air flow shall be horizontal.

WARNING — At the temperature used, organic solvent can form explosive mixtures with air. It is therefore important that the solvent vapour concentration in the oven is not allowed to exceed a value at which an explosion could occur.

For referee tests, ovens of the same design shall be used by all parties.

5.5 Analytical balance, capable of weighing to 0,001 g.

5.6 Desiccator.

6 Sampling

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

Examine and prepare each sample for testing, as described in ISO 1513.

7 Procedure

Carry out the test in duplicate.

Weigh a clean, dry centrifuge tube (5.2) (m_0) to the nearest 0,01 g. Into this centrifuge tube weigh out accurately to the nearest 0,01 g the amount of sample (m_1) indicated in Table 1. Select a suitable solvent or solvent mixture for the coating material with as low a boiling point as possible and add the amount indicated in the table. For example, for solvent-soluble paints use xylene, for water-dilutable paints use butylglycol. Stir the sample with the ball stirrer (5.3) until the binder has dissolved in the solvent. Then rinse any residue adhering to the stirrer quantitatively into the centrifuge tube using the same solvent, ensuring that the tube is filled not more than four-fifths full. Centrifuge until the solid particles have sedimented out and the supernatant binder solution is nearly clear (this will usually take about 20 min).

NOTE It is not always possible to get the supernatant solution clear as this depends on the pigment and the binder system. Difficulties may be encountered with coating materials containing dyestuffs, carbon black, very finely divided silicon dioxide or very finely divided titanium dioxide.

After decanting the supernatant solution, resuspend the solid particles at the bottom of the centrifuge tube in the same solvent as used in the first centrifuging operation. Make up to the mark and centrifuge for a further 20 min, and then proceed as for the first centrifuging operation. Repeat this procedure a third and, if necessary, a fourth time.

After the final centrifuging operation, dry the tube containing the sedimented solids at (125 ± 2) °C to constant mass in the drying oven (5.4). Cool to room temperature in the desiccator (5.6) and weigh the tube containing the residue to the nearest 0,01 g (m_2).

Table 1 — Test portion masses and solvent volumes

Volume of centrifuge tube ml	Mass of test portion g	Approximate volume of solvent ml
50	10 ± 0,2	20
100	15 ± 0,3	40

8 Expression of results

Calculate the pigment content as a percentage by mass using the following equation:

$$\text{Pigment content} = \frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the centrifuge tube;

m_1 is the mass, in grams, of the test portion and centrifuge tube;

m_2 is the mass, in grams, of the residue and centrifuge tube.

If the two results (duplicates) differ by more than 2 % (relative to the mean), repeat the procedure described in clause 7.

Calculate the mean of two valid results (replicates) and report the test result to the nearest 0,1 % by mass.

9 Precision

9.1 General

The precision of the method depends on the pigment content of the product under test. The following values apply to a pigment content of about 30 %.

9.2 Repeatability, *r*

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method may be expected to lie with a 95 % probability is 2 %.

9.3 Reproducibility, R

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method may be expected to lie with a 95 % probability is 4 %.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this part of ISO 14680 (ISO 14680-1);

c) the solvent or solvent mixture used;

d) the result of the test as indicated in clause 8, including the individual values and the mean value;

e) any deviation from the test method specified;

f) the date of the test.

ICS 87.060.10

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