INTERNATIONAL STANDARD

ISO 14680-3

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Paints and varnishes — Determination of pigment content —

Part 3: Filtration method

Peintures et vernis — Détermination de la teneur en pigment

Partie 3: Méthode par filtration

Reference number
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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 14680-3 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 3:
Filtration 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 potassium hydroxide solution is added to coagulate the pigment and the resulting solids filtered off. The method is particularly applicable to coating materials containing carbon black, very finely divided silicon dioxide, very finely divided titanium dioxide, organic pigments or polymer dispersions. It is not applicable to many water-dilutable paints because the whole binder will coagulate when the potassium hydroxide solution is added.

The pigment content of coating materials can also be determined by a centrifuge method (see ISO 14680-1) or by an ashing method (see ISO 14680-2).

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 filtration
the proportion by mass of solid particles which is left behind as a residue on filtration under specified conditions in the product under test

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 it has been diluted with solvent and a solution of potassium hydroxide in methanol has been added, a test portion of the product under test is filtered using a glass filter crucible containing a bed of filtering aid. The solids removed by filtration are dried and weighed. In the case of completely soluble binders, the pigment content is calculated from the mass of the solids and that of the test portion.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 50 ml wide-neck conical flask, with conical socket and fitting polytetrafluoroethylene stopper. The conical socket shall not be greased.

5.2 G3 glass filter crucible (pore size 15 μm to 40 μm).

5.3 Suction flask, with filter adapter and rubber seal.

5.4 5 ml safety pipette.

5.5 50 ml measuring cylinder.

5.6 Drying oven, with forced ventilation, capable of being maintained at (105 ± 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.

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

5.8 Desiccator.

6 Reagents and materials

6.1 Filtering aid, for example diatomaceous earth.

6.2 Potassium hydroxide solution in methanol, c(KOH) = 1 mol/l.

6.3 Methanol, analytical grade.

6.4 Suitable organic solvent.

7 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.

8 Procedure

8.1 Preparation of glass filter crucible

Suspend 10 g of filtering aid (6.1) in 150 ml of distilled water. Pour the suspension into the glass filter crucible (5.2) and remove the water by suction. Dry the glass filter crucible with the bed of filtering aid for 1 h at (105 ± 2) °C in the drying oven (5.6), cool to room temperature in the desiccator (5.8) and weigh to the nearest 0,001 g.

8.2 Determination of pigment content

Carry out the test in duplicate.

Weigh 1 g to 2 g of sample (m_2) to the nearest 0,001 g into the conical flask (5.1) and add immediately 20 ml of a suitable organic solvent from the measuring cylinder (5.5). Then, using the safety pipette (5.4), add 1,5 ml of methanolic potassium hydroxide solution to the contents of the flask and close it with the stopper.

After 5 min, filter the contents of the flask into the weighed glass filter crucible (m_1) prepared as described in 8.1. Rinse any residue in the flask into the crucible using 20 ml of a suitable organic solvent from a measuring cylinder, then wash the solids on the filter with methanol until they are free of alkali. Dry the glass filter crucible to constant mass in the oven at 105 °C, cool it to room temperature in the desiccator and weigh to the nearest 0,001 g (m_3).

9 Expression of results

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

$$\text{Pigment content} = \frac{m_3 - m_1}{m_2} \times 100$$

where

m_1 is the mass, in grams, of the prepared glass filter crucible;

m_2 is the mass, in grams, of the test portion;

m_3 is the mass, in grams, of the prepared glass filter crucible and the solids.

If the two results (duplicates) differ by more than 0,5 % (relative to the mean), repeat the procedure described in clause 8.

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

10 Precision

10.1 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 0,5 %.

10.2 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 1 %.

11 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 14680-3);

c) the organic solvent used;

d) the result of the test as indicated in clause 9, including the individual values and the mean value;

e) any deviation from the test method specified;

f) the date of the test.