
**Paints and varnishes — Determination of
pigment content —**

**Part 2:
Ashing method**

*Peintures et vernis — Détermination de la teneur en pigment —
Partie 2: Méthode par calcination*



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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 14680-2 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 2: Ashing 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 product under test is ashed. The method is not applicable to coating materials containing aluminium, organic pigments and/or dyestuffs, or inorganic pigments treated with organic compounds. Such constituents, which could make up about 10 % of the pigment, will be lost during ashing.

As the state of many pigments and extenders changes under the specified test conditions, it will depend on the composition and the chosen ashing temperature whether the actual pigment content of the product under test can be deduced from the apparent pigment content found by this method.

The method is generally not suitable for UV paints and varnishes or those containing reactive diluents requiring special heating conditions.

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

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

3.1

pigment content, determined by ashing

the proportion by mass of the product under test which is left as a residue after ashing under the specified conditions

NOTE It includes inorganic pigments, extenders and other solid constituents of the product which are not volatile under the test conditions; however, their state may have altered.

4 Principle

After evaporating the solvent in a test portion of the product under test, the residue is ashed either at about 600 °C to 900 °C, or at 450 °C in the presence of carbonates. The presence of carbonate should be determined by some other, qualitative analytical technique. The pigment content is calculated from the mass of the ignition residue and that of the test portion.

NOTE Some binders are not fully ashed at 450 °C. The result obtained at that temperature may therefore have poor repeatability.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 **Porcelain crucible**, tall form.

5.2 **Drying oven**, with forced ventilation, capable of being maintained at (80 ± 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.

5.3 **Crucible tongs**.

5.4 **Muffle furnace or fast incinerator**.

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.

Heat a porcelain crucible (5.1) to constant mass (m_1) in the muffle furnace or fast incinerator (5.4) at a temperature in accordance with clause 4 and, after cooling in the desiccator (5.6), weigh about 2 g of the sample (m_2) to the nearest 0,001 g into it. Place the porcelain crucible in the oven (5.2) at (80 ± 2) °C and heat until all the solvent has evaporated. Then remove it from the oven and place it in the muffle furnace or fast incinerator and heat the latter to the ashing temperature (see clause 4). In most cases, ashing will be complete after 2 h.

Cool the porcelain crucible by placing it in the desiccator for at least 30 min. Weigh it to the nearest 0,001 g, then place it in the muffle furnace or fast incinerator again for 15 min. Cool it to room temperature in the desiccator and weigh it again. Repeat these operations until the mass (m_3) is constant.

8 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 porcelain crucible;

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

m_3 is the mass, in grams, of the porcelain crucible and residue.

If the two results (duplicates) differ by more than 0,5 % (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 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 %.

9.2 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 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 14680-2);
- c) the ashing temperature 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.

