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Paints, varnishes and binders — Determination of MEQ value of water-based coating materials and binders

Peintures, vernis et liants — Détermination de la valeur MEQ des produits de peinture et liants à base d'eau

Reference number
ISO 15880:2000(E)

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Contents

Page

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15880 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

Paints, varnishes and binders — Determination of MEQ value of water-based coating materials and binders

1 Scope

This International Standard specifies a titrimetric method for determining the MEQ (milli-equivalent) value of water-based binders and coating materials, either as an MEQ(acid) value or an MEQ(alkali) value.

In contrast to other values, e.g. the acid value, saponification value or hydroxyl value, the MEQ value is always related to the non-volatile matter content of the binder or coating material.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 3251:1993, *Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes*.

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes — Sampling*.

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
MEQ value
the number of milli-equivalents (MEQ) of acid or base contained in 100 g of the non-volatile matter of a water-based coating material or binder

4 Reagents

4.1 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,1 mol/l, in 95 % by volume ethanol or in water.

4.2 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,1 mol/l, in water.

4.3 Perchloric acid, standard volumetric solution, $c(HClO_4)$ = 0,1 mol/l, in acetic acid.

4.4 Solvent, for dissolving the sample.

The type and volume of solvent used depend on the solubility of the product under test, but allowance for this is made by carrying out a blank test. The solvent shall be inert to the neutralizing agent and shall be such that no precipitation occurs during the titration.

NOTE In most cases, methoxypropanol has been found suitable.

If necessary, an emulsifier may be used in addition to the solvent.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Titration vessel, e.g. conical flask, of suitable size.

5.2 Analytical balance, capable of weighing to an accuracy of 0,1 mg.

5.3 Heating device.

5.4 Magnetic stirrer.

5.5 Potentiometric titration apparatus, i.e. pH-meter or voltage meter fitted with a glass electrode and a reference electrode.

6 Sampling

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

7 Procedure

7.1 General

Carry out the determination in duplicate.

7.2 Test portion

The mass shall be chosen so that the volume of neutralizing agent used is about 10 ml.

7.3 Determination of the non-volatile matter content

Determine the non-volatile matter content as described in ISO 3251.

7.4 Determination of MEQ(acid)

Weigh, to the nearest 0,001 g, a test portion (mass m_1) into the titration vessel (5.1) and dissolve, if necessary using the heating device (5.3) and the magnetic stirrer (5.4), in solvent (4.4). If necessary, cool the solution to room temperature before carrying out the determination. Titrate the solution with potassium hydroxide solution (4.1) till the point of inflection of the titration curve is reached (volume V_1). If the titration curve shows several steps, take that step located in the most alkaline region as the end point.

Carry out a blank determination following the same procedure, but omitting the test portion (volume V_2).

7.5 Determination of MEQ(alkali)

Weigh, to the nearest 0,001 g, a test portion (mass m_2) into the titration vessel (5.1) and dissolve, if necessary using the heating device (5.3) and the magnetic stirrer (5.4), in solvent (4.4). If necessary, cool the solution to room temperature before carrying out the determination. Titrate the solution with hydrochloric acid (4.2) or perchloric acid (4.3) till the point of inflection of the titration curve is reached (volume V_3). If the titration curve shows several steps, take that step located in the most acidic region as the end point. The use of hydrochloric acid is preferred. In some cases, e.g. when testing water-based coating materials, hydrochloric acid can cause precipitation. In such cases, perchloric acid shall be used.

Carry out a blank determination following the same procedure, but omitting the test portion (volume V_4).

8 Expression of results

Calculate the MEQ value, in millimoles per 100 g of non-volatile matter, by the following equations:

$$\text{MEQ(acid)} = \frac{(V_1 - V_2) \times c \times 10\,000}{m_1 \times \text{NV}} \quad (1)$$

$$\text{MEQ(alkali)} = \frac{(V_3 - V_4) \times c \times 10\,000}{m_2 \times \text{NV}} \quad (2)$$

where

V_1 is the volume, in millilitres, of potassium hydroxide solution required for the determination in 7.4;

V_2 is the volume, in millilitres, of potassium hydroxide solution required for the blank determination in 7.4;

V_3 is the volume, in millilitres, of hydrochloric acid or perchloric acid required for the determination in 7.5;

V_4 is the volume, in millilitres, of hydrochloric acid or perchloric acid required for the blank determination in 7.5;

c is the concentration, in moles per litre, of the titrant (= 0,1 mol/l, see 4.1 to 4.3);

m_1, m_2 is the mass, in grams, of the test portion;

NV is the non-volatile matter content, expressed as a percentage by mass (see 7.3).

Report as the final result the mean to three significant figures, but in any case rounded to not more than one decimal place.

9 Precision

9.1 Repeatability limit (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 5 % (relative to the mean of the two test results) for MEQ(acid) and 3 % (relative to the mean of the two test results) for MEQ(alkali).

9.2 Reproducibility limit (R)

The value below which the absolute difference between two single 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 8 % (relative to the mean of the two test results) for MEQ(acid) and 6 % (relative to the mean of the two test results) for MEQ(alkali).

10 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard (ISO 15880);

b) all details necessary for complete identification of the product tested (manufacturer, trade name, batch number, etc.);

c) the result of the test as indicated in clause 8;

d) any deviation from the test method specified;

e) the date of the test.

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