INTERNATIONAL STANDARD

ISO 4325

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Soaps and detergents — Determination of chelating agent content — Titrimetric method

Savons et détergents — Détermination de la teneur en agent séquestrant — Méthode titrimétrique

Reference number
ISO 4325:1990(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 4325 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 4325:1977), of which it constitutes a minor technical revision.

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Soaps and detergents — Determination of chelating agent content — Titrimetric method

1 Scope

This International Standard specifies a method of analysis for the determination of the chelating agent content of detergent compositions and soaps containing not more than 2 % (*m*/*m*) of chelating agent.

Ethylenediaminetetraacetic acid (EDTA) is one of the most widely used chelating agents and the method has been designed principally for determining this compound and its salts. Before determining other chelating agents, or determining ethylenediaminetetraacetic acid in the presence of other chelating agents, the applicability of the method should be confirmed.

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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 607:1980, *Surface active agents and detergents — Methods of sample division.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Principle

A test portion of the sample is dissolved in water, the pH of the solution adjusted to 4,65 and the solution titrated against standard copper(II) sulfate solution using 1-(2-pyridylazo)-2-naphthol as indicator.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water of purity grade 3 as defined in ISO 3696.

4.1 Hydrochloric acid solution, $c(HCl) = 5$ mol/l.

4.2 Acetate buffer solution, pH = 4,65.

Mix equal volumes of acetic acid solution, $c(CH_3COOH) = 0{,}4$ mol/l, and sodium hydroxide solution, $c(NaOH) = 0{,}2$ mol/l.

4.3 PAN indicator, 0,1 % (*m*/*m*) solution of 1-(2-pyridylazo)-2-naphthol in ethanol.

Do not keep this solution longer than 7 days.

4.4 Copper(II) sulfate, standard volumetric solution, $c(CuSO_4) = 0{,}0100$ mol/l.

Weigh out, to the nearest 1 mg, 2,497 g of copper(II) sulfate pentahydrate ($CuSO_4.5H_2O$), of minimum purity 99,5 % (*m*/*m*) and dissolve in water. Quantitatively transfer the solution obtained to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

5 Apparatus

Ordinary laboratory apparatus and

5.1 pH-meter, with electrodes and stirrer.

5.2 Burette, capacity 50 ml, complying with the requirements of class A of ISO 385-1.

6 Sampling

The laboratory sample of soap or detergent shall be prepared and stored in accordance with the instructions given in ISO 607.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 g, 10 g of the laboratory sample into a beaker or conical flask.

7.2 Determination

Add 100 ml of water to the test portion, warm and stir to dissolve. Introduce the electrodes, connected to the previously calibrated pH-meter (5.1), and add hydrochloric acid solution (4.1) until the pH is 4,6 ± 0,5. Raise, rinse and remove the electrodes.

NOTE 1 In most cases, there is no interference by other components of the product, for example fatty acids, but if necessary the fatty acids of soaps may be removed by filtration through a wet filter paper.

Add 5 ml of the acetate buffer solution (4.2) and 0,4 ml of the PAN indicator solution (4.3). Heat to about 60 °C and titrate with the copper(II) sulfate solution (4.4) until the indicator changes from yellow to wine-red.

NOTE 2 The wine-red colour should persist for at least 1 min. A fading end-point suggests that some other chelating agent is present.

8 Expression of results

8.1 Calculation

The chelating agent content, expressed as a percentage by mass of ethylenediaminetetraacetic acid (EDTA), is given by the following formula:

$$\frac{Vc}{10^3} \times 292 \times \frac{100}{m}$$

where

V is the volume, in millilitres, of the copper(II) sulfate solution (4.4) used for the titration;

c is the actual concentration, expressed in moles of $CuSO_4$ per litre, of the copper(II) sulfate solution used;

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

292 is the relative molecular mass of ethylenediaminetetraacetic acid.

8.2 Precision

8.2.1 Repeatability

The maximum difference found between the results of two determinations carried out on the same sample in rapid succession by the same analyst using the same apparatus should not be more than 0,01 % (*m*/*m*) for EDTA contents up to 2 % (*m*/*m*).

8.2.2 Reproducibility

The maximum difference between two results obtained on the same sample, in two different laboratories, should not differ by more than 0,06 % (*m*/*m*) for liquid soaps, for EDTA contents up to 2 % (*m*/*m*), and by 0,04 % (*m*/*m*) for detergents, for EDTA contents up to 2 % (*m*/*m*).

9 Test report

The test report shall include the following information:

a) all information necessary for the complete identification of the sample;

b) the method used (i.e. a reference to this International Standard);

c) the results and the way in which they have been expressed;

d) any operational details which are not specified in this International Standard or in the International Standards to which reference is made, or which are regarded as optional, as well as any incidents liable to have affected the results.

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