International Standard



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Soaps — Determination of content of ethanol-insoluble matter

Savons — Détermination de la teneur en matières insolubles dans l'éthanol

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 673 was developed by Technical Committee ISO/TC 91, $\it Surface\ active\ agents$.

The first edition (ISO 673-1974) had been approved by the member bodies of the following countries :

Argentina Germany, F.R. Romania Austria Hungary South Africa, Rep. of Belgium Ireland Spain Brazil Israel Sweden Canada Japan Switzerland Chile Netherlands United Kingdom Colombia New Zealand Yugoslavia Egypt, Arab Rep. of Poland France Portugal

No member body had expressed disapproval of the document.

This second edition, which supersedes ISO 673-1974, incorporates draft Amendment 1, which was circulated directly to ISO Council for acceptance in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO.

Soaps — Determination of content of ethanol-insoluble matter

1 Scope and field of application

This International Standard specifies a method for the determination of the content of ethanol-insoluble matter in commercial soaps, excluding compounded products.

2 Definition

For the purpose of this International Standard, the following definition applies.

ethanol-insoluble matter: The matter not dissolved by the procedure specified in this International Standard.

NOTES

- 1 The ethanol-insoluble matter corresponds to the additives and foreign matter, of low solubility or insoluble in 95 % (V/V) ethanol, added to soaps, and also to substances in all soap formulations, such as alkali carbonates and chlorides, of low solubility in 95 % (V/V) ethanol.
- 2 The foreign matter may be inorganic (carbonates, borates, perborates, chlorides, sulphates, silicates, phosphates, iron oxides, etc.) or organic (starches, dextrins, caseins, sugars, cellulose derivatives, alginates, etc.).

3 Principle

Dissolution of the soap in ethanol, filtration and weighing of the undissolved residue.

4 Reagent

4.1 Ethanol, 95 % (V/V).

5 Apparatus

Ordinary laboratory apparatus and

- **5.1** Conical flasks, of capacity 500 ml, having ground glass necks.
- **5.2 Reflux condenser**, water-cooled, with a conical ground glass joint at the bottom to fit the conical flasks (5.1).

- 5.3 Water bath.
- **5.4** Oven, capable of being controlled at 103 \pm 2 °C.
- **5.5** Analytical balance, accurate to 0,001 g.

6 Sampling

Procedures for the preparation and storage of the laboratory sample will form the subject of a future International Standard.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, about 5 g of the laboratory sample into a conical flask (5.1).

7.2 Determination

Add 200 ml of the ethanol (4.1) to the test portion (7.1) in the conical flask and connect the reflux condenser (5.2).

Heat to gentle boiling, swirling in order to avoid, as far as possible, material adhering to the bottom of the flask.

Dry the filter paper to be used for the filtration of the insoluble matter in the oven (5.4), controlled at 103 \pm 2 °C, for 1 h. Allow it to cool to ambient temperature in a desiccator and weigh it to the nearest 0,001 g. Place it in a funnel mounted on a second conical flask (5.1).

When dissolution of the soap appears to be complete, decant the supernatant liquid on to the filter paper, wash the insoluble matter in the conical flask by decantation with the ethanol (4.1), previously heated to near its boiling point, and transfer the insoluble matter to the filter paper with the aid of small quantities of warm ethanol (4.1).

Wash the filter paper and the residue with the warm ethanol until entirely free from soap.¹⁾

During this operation, it is advantageous to place the conical flask carrying the funnel on the water bath (5.3) so as to keep the filtrate gently boiling. An independently heated funnel may also be used.

¹⁾ The final washings should show no appreciable residue on evaporation.

At the same time, cover the funnel with a watch glass. By this means, cooling of the wash liquor is avoided and ethanol vapour, which condenses on the watch glass and drops back on the filter paper completes the washing of the latter. Dry the filter paper in air and then place it in the oven (5.4), controlled at 103 \pm 2 $^{\rm o}$ C. After 1 h, remove the filter paper, leave it in the desiccator just long enough for it to cool completely to ambient temperature and weigh it. Repeat the operations of drying in the oven, cooling and weighing until the difference in mass between two successive weighings is less than 0,001 g. Note the final mass.

NOTE — With certain soaps, especially silicated soaps, the insoluble matter cannot be completely detached from the bottom of the conical flask. In this case, after thoroughly washing the residue with ethanol, dissolve it in a little hot distilled water. Transfer this solution to a weighed evaporating dish, evaporate on the boiling water bath and dry in the oven (5.4), controlled at 103 \pm 2 °C. Allow to cool in a desiccator and weigh. Repeat the operations of drying (for periods of 1 h), cooling and weighing until the difference in mass between two successive weighings is less than 0,001 g. Add this mass to that of the residue on the filter paper.

8 Expression of results

8.1 Calculation

The content of ethanol-insoluble matter, expressed as a percentage by mass, is given by the formula

$$\frac{m}{m_0} \times 100$$

where

 m_0 is the mass, in grams, of the test portion;

m is the mass, in grams, of the residue.

8.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories shall not exceed 0,05 % for contents of ethanol-insoluble matter less than or equal to 1 %, and shall not exceed 0,1 % for contents of ethanol-insoluble matter higher than 1 %.

9 Test report

The test report shall include the following particulars:

- a) all information necessary for the complete identification of the sample;
- b) the reference of the method used (reference to this International Standard);
- c) the results, together with the form in which they are expressed;
- d) the test conditions;
- e) any operations not specified in this International Standard, or regarded as optional, as well as any incidents likely to have affected the results.