# INTERNATIONAL STANDARD

# ISO 4318

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## Surface active agents and soaps — Determination of water content — Azeotropic distillation method

*Agents de surface et savons — Détermination de la teneur en eau — Méthode par entraînement azéotropique*



Reference number
ISO 4318 : 1989 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4318 was prepared by Technical Committee ISO/TC 91, *Surface active agents.*

This second editions cancels and replaces the first edition (ISO 4318 : 1978), of which it constitutes a minor revision.





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# Surface active agents and soaps — Determination of water content — Azeotropic distillation method

## 1 Scope

This International Standard specifies an azeotropic distillation method for the determination of the water content of surface active agents and soaps. Volatile matter soluble in water but insoluble in xylene or petroleum will be included in the result.

The method is applicable to products in the form of powders having water contents greater than 5 % ($m/m$), and in the form of pastes and solutions. It is not applicable to samples containing water-soluble volatile compounds, for example ethanol.

With regard to soaps, as the results cannot be obtained with an accuracy greater than 0,3 %, this method should be used only for soaps containing appreciable amounts of volatile matter insoluble in water. The use of this method is also recommended for soaps made with linseed oil or other drying oils and for certain soaps containing, for example, sodium silicate.

The azeotropic distillation method is applicable only if so indicated in the specific standard for each product.



## 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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383 : 1976, *Laboratory glassware — Interchangeable conical ground joints.*

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

ISO 8212 : 1986, *Soaps and detergents — Techniques of sampling during manufacture.*

## 3 Principle

Azeotropic distillation of the water in a test portion with boiling xylene or petroleum.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade.

**WARNING — Xylene being a toxic solvent, the safety instructions for the handling of poisonous solvents must be observed.**

**4.1 Xylene** (any of the isomers or a mixture of the isomers in any proportion), boiling range 130 °C to 140 °C, or

**4.2 Petroleum,** boiling range 180 °C to 220 °C.

## 5 Apparatus

Ordinary laboratory apparatus and:

**Distillation apparatus,** comprising the following items:

**5.1 Flask,** of minimum capacity 500 ml, fitted with a ground glass joint complying with the requirements of ISO 383.

**5.2 Graduated collecting cylinder,** of capacity 2 ml or 10 ml (Dean-Stark receiver).

The graduation interval and its tolerance for the 2 ml graduated collecting cylinder shall be

- graduation in 0,1 ml;
- maximum error: 0,05 ml.

The graduation interval and its tolerance for the 10 ml graduated collecting cylinder shall be, after 1 ml:

- graduation in 0,2 ml;
- maximum error: 0,1 ml.

**5.3 Reflux condenser,** connected to the flask (5.1) and the collecting cylinder (5.2).

Before use, remove all traces of fatty matter from the graduated collecting cylinder (5.2) and the interior tube of the reflux condenser (5.3) by washing them successively with, for example, chromic-sulfuric acid mixture, distilled water and acetone. Then dry them. Perfect cleanliness of the apparatus is essential to the success of the test.

## 6 Sampling

The laboratory sample of the surface active agent or soap shall be prepared and stored in accordance with the instructions given in ISO 607 or ISO 8212.

## 7 Procedure

**WARNING — This procedure must be carried out in a fume cupboard, to prevent the exposure of laboratory workers to toxic xylene vapour if xylene is used rather than petroleum.**

### 7.1 Test portion

Weigh, to the nearest 0,01 g, 10 g to 50 g of the laboratory sample into the flask (5.1), the test portion size being chosen so that the graduated cylinder (5.2) will be at least 50 % full at the end of the test.

### 7.2 Determination

Add to the test portion (7.1), 100 ml to 300 ml of xylene (4.1) or petroleum (4.2) and an anhydrous anti-bumping agent, for example fragments of pumice. Connect the flask to the rest of the apparatus.

Heat gradually to boiling and continue boiling until the xylene or petroleum distilling (2 or 3 drops of reflux per second) is clear and there is no further separation of water.

If drops of water adhere to the wall of the tube, detach them during and after distillation, for example by dislodging them with a wire spiral and washing down with 5 ml of xylene or petroleum.

If foam interferes with the determination, it may be eliminated by adding some dry paraffin or oleic acid to the flask.

Allow to stand until the water has completely separated, and no emulsified zone remains.

Read the volume of water in the graduated tube, at the reference temperature of 20 °C.

## 8 Expression of results

### 8.1 Method of calculation

The water content, expressed as a percentage by mass, is given by the formula

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

where

$V$ is the volume, in millilitres, of the aqueous layer in the graduated tube;

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

### 8.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not exceed 1 % ($m/m$).

## 9 Test report

The test report shall include the following particulars:

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

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

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

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