
**Surface-active agents and detergents —
Determination of water content —
Karl Fischer methods**

*Agents de surface et détergents — Détermination de la teneur en
eau — Méthodes de Karl Fischer*



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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4317 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This third edition cancels and replaces the second edition (ISO 4317:1991), which has been technically revised to incorporate the following changes:

- the coulometric titration method in 5.3 has been added;
- the former Annex B has been deleted;
- interlaboratory test results have been added in a new Annex B;
- the Bibliography has been added.

Surface-active agents and detergents — Determination of water content — Karl Fischer methods

1 Scope

This International Standard specifies two titration methods (volumetric and coulometric) using Karl Fischer reagent for the determination of the water content of surface active agents and detergents.

These methods are applicable to products in the form of powders, pastes and solutions.

They are applicable only if so indicated in the specific standard for each product.

As alkaline compounds react with Karl Fischer reagent, the methods give values which are too high in the case of samples containing alkali metal silicates, carbonates, hydroxides or borates. Therefore, samples need to be analysed for the presence of such alkali metal salts prior to the determination of the water content.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

water content

mass fraction of free water, water of crystallization, absorbed water or occluded water, calculated from the amount of Karl Fischer reagent used in accordance with this International Standard

NOTE The water content is expressed as a percentage by mass.

4 Principle

The water content of a test portion is determined on the basis of the reaction of water with Karl Fischer reagent.

5 Test methods

5.1 General

Two titration methods using Karl Fischer reagent are described: a volumetric titration method and a coulometric titration method.

The volumetric titration method shall be used for samples with a water content of 2 % or more; the coulometric titration method shall be used for samples with a water content of 0,1 % or less. Both methods may be used for water contents in the range of 0,1 % to 2 %.

5.2 Volumetric titration method

5.2.1 Principle

Any water present in a test portion is reacted with a solution of iodine and sulfur dioxide in an appropriate mixture (Karl Fischer reagent), previously standardized by titration with an exactly known mass of water. The water content is calculated as a percentage by mass from the amount of reagent used.

5.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only grade 2 distilled water (5.2.2.1).

5.2.2.1 Distilled water or water of at least equivalent purity, complying with the requirements of ISO 3696 for grade 2.

5.2.2.2 Karl Fischer reagent, preferably commercially prepared.

A reagent containing pyridine may be prepared as described in Annex A for information purposes. A non-pyridine reagent can be used if it has been shown to be suitable; if so, this shall be indicated in the test report.

NOTE A suitable non-pyridine-containing reagent containing iodine, sulfur dioxide and an amine dissolved in 2-methoxyethanol is commercially available.

5.2.2.3 Disodium tartrate dihydrate or **oxalic acid dihydrate**, to be used as the primary standard for Karl Fischer reagent, or **water** (5.2.2.1).

5.2.2.4 Solvent, e.g. 2-methoxyethanol, propan-1-ol or methanol.

If it is known that these solvents interfere with the determination (e.g. for the determination of water in aldehydes or ketones), trichloromethane or dichloromethane may be used; if so, this shall be indicated in the test report.

WARNING — Trichloromethane and dichloromethane are harmful if inhaled or swallowed, and are irritating to the skin, with the risk of irreversible effects and the danger of serious damage to health in the event of prolonged exposure.

5.2.3 Apparatus

Use usual laboratory apparatus and, in particular, the following.

5.2.3.1 Karl Fischer apparatus, fully automatic or semi-automatic, consisting of

- a titrator with double platinum electrode,
- a 20 ml piston burette,