

German standard methods for the examination of water, waste water and sludge

General information (group A)

Pretreatment, aliquotation and homogenization of non-homogeneous water samples for the determination of chemical oxygen demand (COD) (A 30)

DIN**38 402**

Part 30

Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlammuntersuchung; allgemeine Angaben (Gruppe A); Vorbehandlung, Teilung und Homogenisierung heterogener Wasserproben für die Bestimmung des chemischen Sauerstoffbedarfs (CSB) (A 30)

In keeping with current practice in standards published by the International Organization for Standardization (ISO), a comma has been used throughout as the decimal marker.

This standard has been prepared jointly with Study Group *Wasserchemie* of the *Gesellschaft Deutscher Chemiker* (German Chemists' Society) (see Explanatory notes).

It will be necessary to resort to the services of specialists and to make use of specialized laboratories in connection with the examinations specified in this standard.

1 Field of application

This standard describes a standard method for the pretreatment, aliquotation and homogenization of non-homogeneous water samples (with a volume between 250 ml and 20 l) for the subsequent determination of the chemical oxygen demand (COD).

If suspended substances are present, this method does not always ensure a representative sample aliquotation and homogenization. The method is not applicable to water samples which contain several liquid phases (see clause 4).

2 Concepts**2.1 Non-homogeneous water samples**

For the purpose of this standard, non-homogeneous water samples are water samples which contain settleable solids and/or suspended solids.

2.2 Homogenization

For the purpose of this standard, homogenization is the homogenization of a non-homogeneous water sample which is achievable using the method described in this standard (see clause 7).

3 Principle

The water sample is taken with due consideration given to the sampling instructions given in DIN 38 402 Parts 11 to 15, split up if necessary either at the sampling site or in the laboratory into representative subsamples and then homogenized by stirring with a magnetic stirrer under defined conditions (see clause 7). Aliquot amounts necessary for the COD determination are pipetted from the homogenized sample while stirring with a magnetic stirrer.

4 Interference**4.1 Interference during pretreatment**

Representative sampling, sample aliquotation and sample homogenization cannot be carried out where substances are present which form multiphase systems which are

not miscible or sparingly miscible with water. If the non-aqueous phase cannot be separated without the aqueous phase being impaired (entrainment, for example, of solids), further examination using this method is not possible. In the case of separable multiphase systems, the aqueous phase can be separated and examined separately. The analytical result obtained under these conditions can, however, not be regarded as representative of the complete water sample.

4.2 Interference during homogenization and taking of subsamples

Solid particles having a markedly higher density than water (e.g. sand and gravel) settle very rapidly and are usually not included in the determination. Readily volatile substances may outgas and give rise to findings which are too low. Solid particles may completely or partially clog the pipette opening or the sampling valves, and this may result in a filtering effect.

Other solid particles (e.g. cellulose fibres) are not adequately homogenized in the conditions of the method. Magnetisable particles stick to the magnetic stirrer.

In both the last mentioned cases different equipment (see subclause 7.3, note 2) shall be used for homogenizing.

5 Designation

Designation of the method used for pretreatment, aliquotation and homogenization of non-homogeneous water samples for the subsequent determination of chemical oxygen demand (COD) (A 30):

Method DIN 38 402 – A 30

6 Apparatus

All the following apparatus required shall be clean and free from grease and dust when used.

Flat bottom flasks made of glass or plastics, of nominal capacities 250 ml, 5 l and 10 l, e.g.

DIN 12 039 – W 250-G flat bottom flask.

Containers for sampling fairly large volumes, e.g. 20 l.

Conical flasks, of nominal capacity 250 ml, e.g.

DIN 12 380 – EE 250 flask.

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