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

General information (group A)

Pretreatment, homogenization and division of non-homogeneous water samples (A 30)

DIN 38402-30

ICS 13.060.30

Supersedes July 1986 edition.

Descriptors: Water analysis, homogenization, sample pretreatment.

Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlammuntersuchung – Allgemeine Angaben (Gruppe A) – Teil 30: Vorbehandlung, Homogenisierung und Teilung heterogener Wasserproben (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.

Foreword

This standard has been prepared by the *Normenausschuß Wasserwesen* (Water Practice Standards Committee) jointly with Study Group *Wasserchemie* (Water Chemistry) of the *Gesellschaft Deutscher Chemiker* (German Chemists' Society) (see Explanatory notes).

Expert assistance and specialized laboratories will be required to perform the analyses specified in this standard.

Depending on the objective of the analysis, a check shall be made in individual cases as to whether and to what extent additional conditions have to be specified.

Amendments

The following amendments have been made to the July 1986 edition.

a) The scope of the standard has been extended to include samples of volumes up to 30 l.

b) The specifications of the standard have been brought into line with the state of technology.

Previous edition

DIN 38402-30: 1986-07.

1 Scope

This standard specifies a standard procedure for the pretreatment, homogenization and division of non-homogeneous water samples of volumes up to 30 l.

If suspended matter is present, this method does not always ensure representative sample division and homogenization. The method is not suitable for water samples containing several liquid phases (cf. clause 5) and for analyses where sample preparation causes a change in the concentration of the analytes (e.g. when determining dissolved gases or volatile compounds).

2 Normative references

This standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the titles of the publications are listed below. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated into it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

Continued on pages 2 to 10.

Translation by DIN-Sprachendienst.

In case of doubt, the German-language original should be consulted as the authoritative text.

DIN 12039	Wide mouth flat bottom flasks with conical ground glass socket and stopper, for laboratory use
DIN 12331	Beakers for laboratory use
DIN 12380	Narrow mouth conical flasks for laboratory use
DIN 12650-3	Piston-operated dispensers for laboratory use
DIN 12691	Class AS fast delivery one-mark bulb pipettes, with a waiting time of 15 seconds, for laboratory use
DIN 38402-11	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling of waste water (A 11)
DIN 38402-12	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling from stagnant waters (A 12)
DIN 38402-13	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling from aquifers (A 13)
DIN 38402-14	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling of untreated water and drinking water (A 14)
DIN 38402-15	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling from flowing waters (A 15)
DIN V 38402-17	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling from precipitation in or after return to liquid state (A 17)
DIN 38402-18	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling of water from mineral springs and spas (A 18)
DIN 38402-19	German standard methods for the examination of water, waste water and sludge – General information (group A) – Sampling of swimming pool and bathing pool water (A 19)
DIN 38402-42	German standard methods for the examination of water, waste water and sludge – General information (group A) – Evaluation of interlaboratory tests (A 42)
DIN 38405-13	German standard methods for the examination of water, waste water and sludge – Anions (group D) – Determination of cyanides (D 13)
DIN 38409-2	German standard methods for the examination of water, waste water and sludge – Parameters characterizing effects and substances (group H) – Determination of filterable matter and the residue on ignition (H 2)
DIN 38409-9	German standard methods for the examination of water, waste water and sludge – Parameters characterizing effects and substances (Group H) – Determination of the percentage by volume of settleable matter in water and waste water (H 9)
DIN 38409-41	German standard methods for the examination of water, waste water and sludge – Parameters characterizing effects and substances (group H) – Determination of chemical oxygen demand (COD) for COD values above 15 mg/l (H 41)
DIN 38412-31	German standard methods for the examination of water, waste water and sludge – Bioassays (group L) – Determining the tolerance of fish to the toxicity of waste water by way of a dilution series (L 31)
DIN EN 1189	Water quality – Determination of phosphorus by the ammonium molybdate spectrometric method
DIN EN 1483	Water quality – Determination of mercury
DIN EN 1484	Water analysis – Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)
DIN EN 1485	Water quality – Determination of adsorbable organically bound halogens (AOX)
DIN EN 1899-1	Water analysis – Determination of biological oxygen demand after n days (BOD_n) of water – Part 1: Dilution and seeding method with allylthiourea addition (ISO 5815 : 1989, modified)
DIN EN ISO 5961	Water quality – Determination of cadmium by atomic adsorption spectrometry (ISO 5961 : 1994)

[1] Funk, W., Damman, V. and Donnevert, G. *Qualitätssicherung in der Analytischen Chemie* (Quality assurance in analytical chemistry), Weinheim: *VCH Verlagsgesellschaft*, 1992.

3 Concepts

3.1 Non-homogeneous water sample

A water sample containing undissolved matter.

3.2 Sample preparation (of non-homogeneous water samples)

All the working steps needed to prepare the sample for analysis, including pretreatment, homogenization and division and, if required, preservation, digestion, enrichment and dilution.

3.3 Sample pretreatment (of non-homogeneous water samples)

Removal of non-homogenizable components.

3.4 Homogenization of a water sample

Treatment of a non-homogeneous water sample, using a specified homogenization method, to achieve uniform distribution of dissolved and undissolved components.

NOTE: See clause 8 for homogenization methods.

3.5 Sample division

3.5.1 Taking of subsamples

Dividing a sample into a number of subsamples of unspecific size.

3.5.2 Taking of aliquots

Withdrawal of measured amounts (aliquots) from the original sample, a subsample or a dilution thereof.

4 Principle

The water sample is taken, with due consideration given to the sampling specifications in DIN 38402-11 to DIN 38402-19, and homogenized and divided under defined conditions as specified in clause 8, either at the sampling site or in the laboratory.

5 Interferences

5.1 General

Representative sampling, sample division and sample homogenization are impossible in the presence of liquids that form multiphase systems which are insufficiently water-miscible.

If the non-aqueous phase cannot be removed without adversely affecting the aqueous phase (e.g. by entrainment of undissolved matter), sample treatment by this method is not possible. If the multiphase system is separable, the aqueous phase can be removed and analysed separately. However, the analytical result then obtained will not be representative of the water sample (note this in the test report).

5.2 Interference during homogenization and sample division

Undissolved matter having a significantly higher density than water (e.g. sand and gravel) settles very rapidly and is not usually included in the determination. Volatile substances may outgas, which can lead to erroneously low results. Undissolved matter may partially or completely clog the pipette orifice or sampling valves, thus producing an unwanted filtration effect.

6 Designation

Designation of the method used for pretreatment, homogenization and division of non-homogeneous water samples (A 30):

Method DIN 38402 – A 30

7 Apparatus

All equipment used shall be clean and free from grease and dust. In addition to standard glass or plastic laboratory vessels of a range of capacities not exceeding 30 l, the following equipment shall be used.

a) **Flat bottom flasks** (e.g. DIN 12039 – W 250-G flat bottom flasks).
b) **Conical flasks** (e.g. DIN 12380 EE 250 flasks).
c) **Beakers** (e.g. DIN 12331 – H 250 beakers).
d) **Magnetic stirrer**, with speed control, and a magnetic follower of suitable length.
e) **Stirrer motor**, with variable speed control and blade stirrers.
f) **Ultrasonic bath** (e.g. with a power rating of 240 W).
g) **Ultrasonic probe**.
h) **Dispersing tool**[1]), rated for speeds up to 20 000 rpm.
i) **One-mark bulb pipettes**, of nominal capacities 10, 20 and 50 ml (e.g. DIN 12691 – VPAS 10 one-mark bulb pipettes).
j) **Dispenser**, of nominal capacity 20 ml (e.g. DIN 12650-3 dispenser).

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[1]) Information on sources of supply is obtainable from *Normenausschuß Wasserwesen im DIN*, D-10772 Berlin.

8 Procedure

8.1 Sample pretreatment

Examine the water sample for non-homogenizable components (cf. clause 5) and remove matter such as plant parts and water-borne organisms which are not representative of the water sample.

8.2 Homogenization

8.2.1 Shaking

Samples having a volume of less than 5 l may be shaken by hand, provided that this ensures sufficient mixing.

8.2.2 Stirring

Magnetic stirrers or blade stirrers may be used.

If blade stirrers are used, ensure that the blades are of sufficient size and are immersed as deeply as possible.

If magnetic stirrers are used, ensure that the magnetic follower is large enough (about 1/3 of the vessel diameter). It has sometimes proved advantageous to use followers with lateral vanes.

Adjust the stirrer speed to give a vortex of about 10 % of the liquid depth; it will sometimes be advisable to change the direction of stirring at short intervals (alternating stirring). In exceptional cases, for subsampling, the sample may be stirred by hand in situ.

8.2.3 Using an ultrasonic bath

Place the covered sample vessels in the bath and adjust the filling height of the water bath to that of the sample vessel.

Homogenize with sufficient power and duration (e.g. about ten minutes for a power rating of 240 W). The sample may heat up during homogenization; cool it if necessary.

With some samples, the ultrasonic bath treatment does not lead to homogenization, but to size reduction of undissolved matter. In such cases, homogenize samples by shaking or stirring before or during sample division.

8.2.4 Using an ultrasonic probe

Immerse a probe, of size appropriate to the sample volume (follow the manufacturer's instructions) in the sample vessel. Homogenize the sample for a period which will depend on the sample volume used (e.g. sonicate a 100 ml sample for 60 seconds at 350 W). The sample may heat up during homogenization; cool it if necessary.

8.2.5 Using a dispersing tool

Immerse a dispersing tool of a size appropriate to the sample volume (follow the manufacturer's instructions) in the sample vessel. Homogenize the sample for a period which will depend on the volume used (e.g. a 500 ml sample for 60 seconds at 20 000 rpm).

The sample may heat up during homogenization; cool it if necessary. Intense foaming or flotation of undissolved matter may lead to erroneous results.

8.3 Sample division

8.3.1 General

The method of homogenization used will depend on the initial sample volume, the size, type and quantity of particles in the sample, and the parameters to be determined.

To keep the scatter of results as small as possible, analyse as large a subsample volume as possible. Table 1 outlines possible or preferable combinations.

8.3.2 Subsampling

Homogenize samples by shaking, as specified in subclause 8.2.1, or by stirring, as specified in subclause 8.2.2, taking the subsamples during or immediately after homogenization.

8.3.3 Taking of aliquots

When taking aliquots, only shake samples or subsamples by hand if adequate mixing is ensured.

Homogenization by stirring (cf. subclause 8.2.2) will generally be sufficient. If the sample is visibly non-homogeneous even after stirring for a prolonged period, use another homogenization method, taking into account the procedure described below.

First homogenize the sample using an ultrasonic bath. If this is not sufficient, use the ultrasonic probe for small samples (with a volume not exceeding 200 ml) and the dispersing tool for larger samples (with a volume above 200 ml).

Withdraw subsamples during homogenization.

Transfer small volumes (not exceeding 100 ml) to a measuring cylinder using a pipette, and higher volumes via a drainage stopcock or siphon.