SLOVENSKI STANDARD
SIST EN 872:2005

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Kakovost vode - Doloļ evanje suspendiranih snovi ï Metoda s filtracijo skozi filtre iz steklenih vlaken

Water quality - Determination of suspended solids - Method by filtration through glass fibre filters

Wasserbeschaffenheit - Bestimmung suspendierter Stoffe - Verfahren durch Abtrennung mittels Glasfaserfilter

Qualité de l'eau - Dosage des matieres en suspension - Méthode par filtration sur filtre en fibres de verre

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13.060.60	Preiskava fizikalnih lastnosti vode	Examination of physical properties of water

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Water quality - Determination of suspended solids - Method by filtration through glass fibre filters

Qualité de l'eau - Dosage des matières en suspension - Méthode par filtration sur filtre en fibres de verre

Wasserbeschaffenheit - Bestimmung suspendierter Stoffe - Verfahren durch Abtrennung mittels Glasfaserfilter

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Contents

Foreword

This document (EN 872:2005) has been prepared by Technical Committee CEN/TC 230 “Water analysis”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2005, and conflicting national standards shall be withdrawn at the latest by August 2005.

This document supersedes EN 872:1996.

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

The evacuation of large glass vessels can cause dangerous implosions if the vessel is damaged by scratches etc. It should be ensured that the relevant safety precautions have been observed.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document describes a method for the determination of suspended solids in raw waters, waste waters and effluents by filtration through glass fibre filters. The lower limit of the determination is about 2 mg/l. No upper limit has been established.

Water samples are not always stable which means that the content of suspended solids depends on storage time, means of transportation, pH value and other circumstances. Results obtained with unstable samples need to be interpreted with caution.

Floating oil and other immiscible organic liquids will interfere (see Annex A).

Samples containing more than about 1 000 mg/l of dissolved solids can require special treatment (8.6).

NOTE 1 The result of the determination depends to some extent on the type of filter used (5.2). It is therefore recommended that the type of filter is specified.

NOTE 2 The size distribution of particles in different samples can vary widely. Therefore there is no correlation between results obtained with filters of different pore width and no conversion factor can be given for the conversion of results obtained with one type of filter to another.

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.

EN 25667-2:1993, *Water quality — Sampling — Part 2: Guidance on sampling techniques (ISO 5667-2:1991)*.

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:2003)*.

3 Terms and definitions

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

3.1
suspended solids
solids removed by filtration under specified conditions

3.2
dissolved solids
substances remaining, after filtration and evaporation to dryness of a sample, under specified conditions

[4.25.1 of ISO 6107-2:1997]

4 Principle

Using a vacuum or pressure filtration apparatus the sample is filtered through a glass fibre filter. The filter is then dried at 105 °C ± 2 °C and the mass of the residue retained on the filter is determined by weighing.

5 Apparatus

5.1 Equipment for vacuum or pressure filtration, to accommodate the selected filters (5.2).

Equipment for membrane filtration can in most cases be used for other types of filters. The plate supporting the filter needs to have sufficient permeance to allow the water to pass freely.

5.2 Borosilicate glass fibre filters

5.2.1 The borosilicate filters shall satisfy the following criteria

- shall not contain any binders;
- shall be circular and of the appropriate diameter to fit the filtering device (5.1);
- shall comprise a mass per unit area of between 50 g/m^2 and 100 g/m^2;
- the loss of mass obtained in a blank test (5.2.2) shall be less than or equal to 0,017 mg/cm^2 (for the more common diameter of 47 mm, this corresponds to a loss of mass less than or equal to 0,3 mg).

5.2.2 Blank test

Check the loss of mass during filtration by running the procedure in Clause 8 but using 150 ml of distilled water instead of the sample. Check each box or batch of filters separately. Carry out the test 3 times, selecting 3 filters at random, to increase the sensitivity of the test.

NOTE To remove water-soluble constituents, the filters may be prewashed. Individual or a small number of filters (less than 10) are prewashed by filtering 150 ml of distilled water through the filter(s) and then drying at 105 °C for at least 1 h.

It is recommended to bulk-wash filters by soaking in distilled water for several hours. The wash water is drained off and the filter dried at 105 °C for at least 1 h or preferably overnight before use.

Glass fibre filters from different manufacturers can have somewhat different filtering characteristics. State the type of filter used and its manufacturer in the report (see Clause 10).

5.3 Drying oven, capable of maintaining a temperature of 105 °C ± 2 °C.

5.4 Analytical balance, capable of weighing to an accuracy of at least 0,1 mg.

5.5 Drying support of suitably surfaced material, to support the filters in the drying oven (5.3), e.g. Petri dishes.

6 Reagents

6.1 Reference suspension, microcrystalline cellulose, 500 mg/l.

Weigh 0,500 g (oven dry basis) of microcrystalline cellulose $(C_6H_{10}O_5)_n$, the grade used for thin layer chromatography (TLC), or equivalent, and transfer it quantitatively into a 1 000 ml volumetric flask and make up to the mark with distilled water. Shake the suspension well before use.

The suspension may be stored for at least three months.

NOTE The dry matter content of the microcrystalline cellulose can be determined by drying a separate sample in an oven at 105 °C ± 2 °C.

6.2 Working reference suspension, ρ = 50 mg/l.

Shake the reference suspension (6.1) until it is completely uniform. With a minimum delay measure out 100 ml ± 1 ml into a 100 ml volumetric flask. Transfer the measured volume quantitatively into a 1 000 ml volumetric flask and make up to the mark with distilled water. Shake the suspension well before use. Prepare a fresh reagent daily.

7 Sampling and sample handling

Obtain samples as described in the relevant sampling guidelines, such as EN 25667-2 and EN ISO 5667-3. Samples shall preferably be taken in bottles of transparent material. Avoid filling the bottles completely to allow efficient mixing by shaking the bottle.

Analyse samples for the determination of suspended solids as soon as possible after sampling, preferably within 4 h. Store samples which cannot be analysed within 4 h in the dark at between 1 °C and 5 °C, but do not allow the sample to freeze. Interpret results obtained for samples that have been stored more than 2 days with caution. Samples for the determination of suspended solids shall not be preserved by any additives.

If the time period from sampling to analysis exceeds 2 days, this shall be stated in the report as well as the conditions of storage.

8 Procedure

8.1 Allow the samples to attain room temperature.

8.2 Ensure that the filters fulfil the requirements given in 5.2.1.

8.3 Allow a filter to attain moisture equilibrium with the air near the balance and weigh it to the nearest 0,1 mg using the balance (5.4). Take care to avoid dust contaminating the filter, for example by using a desiccator.

8.4 Place the filter, the smooth side down, in the funnel of the filtering device (5.1) and connect the device to a vacuum (or pressure) line.

8.5 Shake the sample bottle vigorously and immediately transfer in one stroke a suitable volume of sample to a measuring cylinder.

If the sample is obtained in a completely filled bottle, mix the sample by transferring it "back and forth" between two bottles. Check that the second bottle is dry and free from contaminants before use.

Select the sample volume so that the dry residue on the filter will be in the optimum range of the determination, which is between 5 mg and 50 mg. However, avoid sample volumes exceeding 1 l. To be valid, the result shall be based on a dry residue of at least 2 mg. Read the sample volume with an accuracy of 2 % or better. Sample volumes of less than 25 ml shall be determined by weighing.

8.6 Filter the sample and rinse the measuring cylinder with about 20 ml of distilled water and use this portion to wash the filter. Rinse the inner sides of the funnel with another 20 ml portion of distilled water.

If the sample contains more than 1 000 mg/l of dissolved solids, repeat the washing of the filter with 3 portions each consisting of 50 ml of distilled water. Take care to wash the rim of the filter.

NOTE The filtering normally is complete within less than 1 min. However, some types of waters contain materials that block the filter pores or reduce their width. This increases the filtering time and the results can become a function of the sample volume. If such blocking of the filter is observed, the determination should be repeated with smaller volumes. The results should be interpreted with caution.

Release the vacuum (or pressure) when the filter is almost dry. Carefully remove it from the funnel with a pair of forceps having flat ends. The filter may be folded if desired. Place the filter on the drying support (5.5) and dry it in the oven (5.3) at 105 °C ± 2 °C for at least 1 h and with a maximum of 14 h to 16 h. Remove from the oven and allow the filter to attain equilibrium with the air surrounding the balance and weigh it as before.

9 Control

Repeat the test procedure (see Clause 8) using 200 ml of the working reference suspension (6.2) as the sample. The recovery shall be between 90 % and 110 %.

10 Calculation and report

10.1 Calculation

Calculate the content of suspended solids from the expression

$$\rho = \frac{1\,000 \times (b - a)}{V}$$

where

ρ is the content of suspended solids, in milligrams per litre, mg/l;

b is the mass of the filter after the filtration, in milligrams, mg;

a is the mass of the filter before the filtration, in milligrams, mg;

V is the volume of the sample, in millilitres, ml. If the sample has been weighed, consider 1 g as equivalent to 1 ml.

10.2 Report

Report results below 2 mg/l as "below 2 mg/l" and other results in milligrams per litre with two significant figures.

The test report shall refer to this document and include the following:

a) date and place of testing;

b) identification mark of the sample tested;

c) the manufacturer and the designation of the filter used;

d) the result;

e) any deviation from the procedure described in this document or any other circumstances that can have affected the results, for example blocking of filters (see the Note in 8.6) and storage time before analysis.

11 Precision

The precision of data for the content of suspended matter, determined as specified in this document, depends mainly on the nature of the sample rather than on the method itself. Furthermore, some influence from the particular make of filter used cannot be excluded.

No generally valid data for the reproducibility of the results can be given since it is virtually impossible to perform an interlaboratory study using authentic waters of relevant types with a guarantee that the subsamples are identical at the arrival in different laboratories. Samples containing living organisms or slimy material (for example carbohydrate polymers) that block filters are particularly sensitive to transport and testing conditions.

In an interlaboratory study (Swedish Environmental Protection Agency, 1992) 175 laboratories analysed two samples by a method, essentially the same as in this document. Synthetic samples prepared with kaolin and microcrystalline cellulose were used. The results summarized in Table 1 illustrate the repeatability: