INTERNATIONAL STANDARD

ISO 11923

First edition
1997-02-01

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

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

Reference number
ISO 11923:1997(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11923 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

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

1 Scope

This International Standard 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 approximately 2 mg/l. No upper limit has been established.

NOTE 1 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 factors. Results obtained with unstable samples need to be interpreted with caution.

Floating oil and other immiscible organic liquids will interfere (see annex B).

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

NOTE 2 The result of the determination depends to some extent on the type of filter used, see 5.2. It is therefore necessary that the type of filter be specified.

NOTE 3 The size distribution of particles in different water samples can vary widely. Therefore there is no correlation between results obtained with filters of different pore diameter, 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 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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 6107-2:—[1], *Water quality — Terminology — Part 2.*

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 suspended solids: Solids removed by filtration or centrifuging under specified conditions [ISO 6107-2:1989, 4.24.3].

3.2 dissolved solids: The substances remaining, after filtration and evaporation to dryness of a sample, under specified conditions [ISO 6107-2:1989, 4.24.1].

---

[1] To be published. (Revision of ISO 6107-2:1989)

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 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).

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

5.2 Borosilicate glass-fibre filters which contain no binders. The filters shall be circular and of the appropriate diameter to fit the filtering device (5.1).

The loss of mass in a blank test shall be less than 0,3 mg per filter. Preferably the areic mass should be between 50 g/m^2 and 100 g/m^2.

Check the loss of mass during filtration by running the procedure in clause 8, but using 150 ml of distilled water instead of the test sample. Check each box or batch separately. Use three filters, selected 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 (< 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.

Filters may be bulk-washed 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 test report (clause 12).

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).

6 Reagents

6.1 Reference suspension of microcrystalline cellulose, $\rho = 500$ mg/l.

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

The suspension has a shelf life of at least three months.

Shake the suspension well before use.

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 cellulose suspension, $\rho = 50$ mg/l.

Shake the reference suspension (6.1) until it is completely uniform. With minimum delay measure (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 working reference cellulose suspension daily.

7 Sampling and sample handling

Obtain samples as described in the relevant sampling guidelines, such as ISO 5667-2. 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 below 8 °C, but do not allow the sample to freeze. Interpret results obtained for samples that have been stored more than 24 h with caution. Samples for the determination of suspended solids shall not be preserved by the addition of any additives.

If the time period from sampling to analysis exceeds 4 h, this shall be stated in the test 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 requirement that the mass loss be less than 0,3 mg per filter (see 5.2).

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

WARNING — The evacuation of large glass vessels can cause dangerous implosions if the vessel is damaged by scratches etc. Ensure that the relevant safety precautions have been observed.

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 contained 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 mass range for the determination, which is 5 mg to 50 mg. However, avoid sample volumes exceeding 1 litre. 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, rinse the measuring cylinder with approximately 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 three 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 water contain materials that block the filter pores or reduce their diameter. 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 the filter 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 1 h to 2 h. Remove the filter from the oven, allow it to attain equilibrium with the air surrounding the balance, and weigh it as before.

9 Control run

Repeat the test procedure (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

Calculate the content of suspended solids ρ, in milligrams per litre, from the equation

$$\rho = \frac{1000(b-a)}{V}$$

where

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

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

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

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

11 Precision

The precision of data for the content of suspended matter, determined as specified in this International Standard, 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 blocks filters are particularly sensitive to transport and testing conditions.

Details of two interlaboratory tests on the precision of the method are summarized in annex A. The values derived from these interlaboratory tests may not be applicable to concentration ranges and matrices other than those given.

12 Test report

The test report shall include reference to this International Standard and the following particulars:

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 departure from the procedure (see clause 8) described in this International Standard or any other circumstances that may have affected the results, for example blocking of filters (see the note to 8.6) and storage time before analysis.

Annex A
(informative)
Results of interlaboratory trials

In an interlaboratory study (Swedish Environmental Protection Agency 1992), 175 laboratories analysed two samples by a procedure essentially the same as in this International Standard, using Whatman GF/A filters. Synthetic samples prepared with kaolin and microcrystalline cellulose were used. The results, summarized in table A.1, illustrate the repeatability:

Table A.1 — Results from an interlaboratory trial in Sweden

Statistical characteristic	Sample 1	Sample 2
Number of accepted results	171	172
Mean	961 mg/l	790 mg/l
Standard deviation	41 mg/l	36 mg/l
Coefficient of variation	4,29 %	4,72 %
Rejected results	4	3

In a German study, reported in 1994, the results in table A.2 were obtained:

Table A.2 — Results from an interlaboratory trial in Germany

Sample	No. of laboratories	Accepted results	Rejected results	Mean mg/l	Coefficient of variation %
Microcrystalline cellulose, 10 mg/l	8	32	0	9,95	9,0
Microcrystalline cellulose, 100 mg/l	8	32	0	96,6	7,8
Microcrystalline cellulose, 500 mg/l	7	26	6	499,5	3,5
Kaolin, 10 mg/l	8	30	2	9,59	7,8
Kaolin, 100 mg/l	8	32	0	92,4	7,5
Kaolin, 500 mg/l	8	31	1	463,4	6,0
Fibrous cellulose (pulp), 10 mg/l	7	28	0	10,3	8,3
Fibrous cellulose (pulp), 100 mg/l	6	24	4	102,3	9,2
Fibrous cellulose (pulp), 500 mg/l	7	28	0	474,1	13,6
Microcryst. cellulose: kaolin, 1: 1, 10 mg/l	8	31	1	9,6	4,4
Microcryst. cellulose: kaolin, 1: 1, 50 mg/l	8	30	2	49,9	1,7
Microcryst. cellulose: kaolin, 1: 1, 200 mg/l	8	32	0	195,4	3,3

The results illustrate the reproducibility that can be expected under ideal conditions, i.e. with samples containing inert particles of well-defined sizes. However, ideal conditions can rarely be expected with authentic samples.

Annex B
(informative)
Samples containing oil or other organic liquids

Oil or other immiscible organic liquids may be retained on the filter and only partially volatilized on drying at 105 °C. Where, however, immiscible oil is important and is to be determined separately, the filtered, water-washed residue should be freed from oil. This may be done by washing first with ethanol and then with hexane before drying at 105 °C. When this procedure is applied, it should be recorded with the test results since some material, other than immiscible oil, could have been extracted.

This page intentionally left blank