SLOVENSKI STANDARD
SIST EN 1899-2:2000

01-januar-2000

Kakovost vode – Določevanje biokemijske potrebe po kisiku po n dneh (BPKn) - 2. del: Metoda za nerazredčene vzorce (ISO 5815:1989, spremenjen)

Water quality - Determination of biochemical oxygen demand after n days (BODn) - Part 2: Method for undiluted samples (ISO 5815:1989, modified)

Wasserbeschaffenheit - Bestimmung des biochemischen Sauerstoffbedarfs nach n Tagen (BSBn) - Teil 2: Verfahren für unverdünnte Proben (ISO 5815:1989, modifiziert)

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Qualité de l'eau - Détermination de la demande biochimique en oxygene apres n jours (DOBn) - Partie 2: Méthode pour les échantillons non dilués (ISO 5815:1989, modifiée)

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ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

SIST EN 1899-2:2000 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD
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EN 1899-2

March 1998

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Descriptors: water tests, water, chemical analysis, determination of content, oxygen demand, samples

English version

Water quality - Determination of biochemical oxygen demand after n days (BOD_n) - Part 2: Method for undiluted samples (ISO 5815:1989, modified)

Qualité de l'eau - Détermination de la demande biochimique en oxygène après n jours (DOB_n) - Partie 2: Méthode pour les échantillons non dilués (ISO 5815:1989, modifiée)

Wasserbeschaffenheit - Bestimmung des Biochemischen Sauerstoffbedarfs nach n Tagen (BSB_n) - Teil 2: Verfahren für unverdünnte Proben (ISO 5815:1989, modifiziert)

This European Standard was approved by CEN on 22 February 1998.

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by September 1998, and conflicting national standards shall be withdrawn at the latest by September 1998.

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

This draft European Standard consists of the following Parts:

EN 1899-1 Water quality – Determination of biochemical oxygen demand after n days (BOD_n) – Part 1: Dilution and seeding method with allylthiourea addition

EN 1899-2 Water quality – Determination of biochemical oxygen demand after n days (BOD_n) – Part 2: Method for undiluted samples

Annex A, which is normative, concerns alternative incubation periods.

Annex B, which is normative, concerns modifications for specific evaluations of the quality of waters.

Annex C, which is informative, contains bibliography.

Endorsement notice

The text of the International Standard ISO 5815:1989 was approved by CEN as a European Standard with agreed common modifications as given below:

- Change in setup (splitting into two parts)
- Additions of annexes.

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Introduction

This European Standard EN 1899-2 is intended for analysis of BOD in waters with a BOD in the range (0,5 to 6) mg/l of oxygen.

The times of incubation specified in the proposal are 5 days - as in ISO 5815:1989, "Water Quality - Determination of biochemical oxygen demand after 5 days (BOD_5) - Dilution and seeding method". Incubation for 5 days - as has been applied in many European countries - or 7 days as has been applied in several Nordic countries through the past many years. The 7 day incubation will typically give higher BOD results than 5 days incubation time.

With an incubation time of 5 days weekend work can only be avoided, if samples are collected Wednesdays, Thursdays to Fridays. With an incubation time of 7 days, samples collected on the first five weekdays can be analysed without implying weekend work. For this reason a 7 day incubation can be considered more convenient than the conventional 5 day incubation.

A modified 7 day incubation is described in a normative annex. The first investigations indicate that BOD results obtained by this modified method are identical to results obtained by the 5 day method described in the proposed standard. It is hoped that more comparative data on these two incubation methods will be obtained during the coming years, so that the modified 7 day incubation method can be included fully at the time of review and revision of this standard.

1 Scope

This European Standard specifies determination of the biochemical oxygen demand of waters of undiluted samples.

This standard is applicable to all waters having biochemical oxygen demands greater than or equal to the limit of determination 0,5 mg/l of oxygen and not exceeding 6 mg/l of oxygen. In this standard, the limit of determination, D_L, is defined as

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$$D_L = t_{0,95(f)} \cdot 2 \cdot s_B \cdot \sqrt{1 + \frac{1}{n}} \qquad (1)$$

where s_B is the within series standard deviation, $t_{0,95(f)}$ is the Student t-value, with f is the degrees of freedom for the determination of s_B and n is the number of analysis for determination of the blank in an analytical series. s_B is calculated from determinations of real samples with a BOD contentration near the estimated D_L.

In cases where the analytical method does not require any blank correction, the term

$$\sqrt{1 + \frac{1}{n}} \qquad (2)$$

is omitted.

The results obtained are the product of a combination of biochemical and chemical reactions. They do not have the rigorous and unambiguous character of those resulting from, for example, a single, well-defined, chemical process. Nevertheless, they provide an indication from which the quality of waters can be estimated.

The test can be influenced by the presence of various substances. Those which are toxic to microorganisms, for example bactericides, toxic metals or free chlorine, will inhibit biochemical oxidation. The presence of algae or nitrifying microorganisms can produce high results. In these situations a modification of the method (see annex B or as described in EN 1899-1.

It is absolutely essential that test conducted according to this standard are carried out by suitably qualified staff.

Annex A describes alternative incubation periods.

Annex B describes procedures for modification of the method by addition of seeding material, salts, inhibition of nitrification by allylthiourea (ATU) addition, neutralisation, homogenisation and/or filtration. These modifications may be found necessary for specific evaluations of the water quality of receiving waters.

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2 Normative references

This European 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 publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3696	Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)
EN 25813	Water quality – Determination of dissolved oxygen – Iodometric method (ISO 5813:1983)
EN 25814	Water quality – Determination of dissolved oxygen – Electrochemical probe method (ISO 5814:1990)
ISO 6107-2:1997	Water quality – Vocabulary – Part 2

3 Definition

For the purposes of this European Standard, the following definition applies:

biochemical oxygen demand after n days (BOD_n): The mass concentration of dissolved oxygen consumed under specified conditions by the biochemical oxidation of organic and/or inorganic matter in water.
n is the incubation time; it is equal to 5 or 7.

NOTE: For the purpose of this European Standard, "biochemical oxidation" is taken to mean "biological oxidation". This definition is similar to the definition of ISO 6107-2.

4 Principle

Equilibration of the sample of water to be analyzed to 20 °C followed by, if necessary, aeration. Incubation at 20 °C for a defined period, 5 or 7 days, in the dark, in a completely filled and stoppered bottle. Determination of the dissolved oxygen concentration before and after incubation. Calculation of the mass of oxygen consumed per litre of sample.

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5 Apparatus

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5.1 Incubation bottles, BOD bottles, with glass stoppers,

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For example, preferably 250 ml to 300 ml or 100 ml to 125 ml, with stoppers and preferably with straight shoulders, or any equivalent bottles.

It is important that the bottles are thoroughly cleaned before use. If the iodometric method (in accordance with EN 25813) for determining dissolved oxygen is used, it is, for example, normally sufficient to rinse the bottle several times with tap water then deionised water. However, if the electrode method (in accordance with EN 25814) is used, a more stringent cleaning procedure, for example, as follows, is required. Add to the empty bottle 5 ml to 10 ml of a wash solution (for example 2,5 g of iodine plus 12,5 g of potassium iodide per litre of 1% (*V/V*) sulfuric acid) shaking well to coat the bottle walls. Let stand for 15 min, pour off the solution and rinse thoroughly with tap water and finally deionised water.

5.2 Incubator, capable of being maintained at (20 ± 1) °C.

5.3 Equipment for determining dissolved oxygen concentration

In accordance with EN 25813 and EN 25814.

5.4 Means of refrigeration, (0 to 4) °C, for transport and storage of the sample.

5.5 Aeration equipment

A bottle of compressed air or a compressor: The air quality shall be such that the aeration does not lead to any contaminations, especially by the addition of organic matter, oxidizing or reducing materials, or metals. If a contamination is suspected, the air shall be filtered or washed.

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6 Storage of the sample

Store the sample at a temperature (0 to 4) °C in a filled and hermetically stoppered bottle immediately after sample collection and until the analysis is performed. Begin the determination of the BOD_n as soon as possible and within 24 h after completion of sample collection.

7 Procedure

7.1 Preparation of test solutions

Bring the test sample to a temperature of (20 ± 2) °C and aerate if necessary. In case of aeration, let the sample stand about 15 min. Remove air bubbles and possible supersaturation of oxygen.

7.2 Determination

7.2.1 Determination via measurement of dissolved oxygen-iodometric method (in accordance with EN 25813)

Using each sample (see 7.1), fill two incubation bottles (5.1) allowing them to overflow slightly. During filling operations, precautions shall be taken to prevent changing the oxygen concentration of the medium.

Allow any air bubbles adhering to the walls to escape. Stopper the bottles, taking care to avoid trapping air bubbles.

Divide the bottles into two series, each containing one bottle of each sample.

Put one series of bottles (the first) in the incubator (5.2) and leave in darkness for n days ± 4 h.

In the second series of bottles measure the dissolved oxygen concentration in each of the bottles at time zero after 15 min, using the method specified in EN 25813 with the addition of azide in the alkaline iodide-azide reagent.

After the incubation, determine the dissolved oxygen concentration in each of the first series of bottles, using the method specified in EN 25813.

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7.2.2 Determination via measurement of dissolved oxygen-electrochemical probe method (in accordance with EN 25814)

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Using each sample (see 7.1) fill an incubation bottle (5.1), allowing it to overflow slightly. During filling operations, precautions shall be taken to prevent changing the oxygen concentration of the medium.

Allow any air bubbles adhering to the walls to escape.

Measure the dissolved oxygen concentration in each of the bottles at time zero, using the method specified in EN 25814.

Stopper the bottles, taking care to avoid trapping air bubbles.

Put the bottles in the incubator (5.2) and leave in darkness for n days ± 4 h.

After the incubation, determine the dissolved oxygen concentration in each of the bottles, using the method specified in EN 25814.

7.2.3 Control analysis

For each series of determinations include at least one double determination of a sample (BOD_{n1}, BOD_{n2}).

Plot the relative difference (r_i) of each series (i) on quality control charts:

$$r_i = \frac{(BOD_{n1} - BOD_{n2}) \cdot 100}{1/2\ (BOD_{n1} + BOD_{n2})}\ \% \qquad (3)$$

where:

BOD_{n1} first BOD_n determination of sample;

BOD_{n2} second BOD_n determination of sample.

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Consider the upper control limit as:

$$3{,}2678 \cdot \bar{r}\ \% \qquad (4)$$

where:

$\bar{r}$ is the average value of r_i-values.

The repeatability variation coefficient (CV_R) can be calculated as:

$$CV_R = \frac{\bar{r}}{1{,}128}\ \% \qquad (5)$$

NOTE 1: After incubation, the residual dissolved oxygen concentration should be between the limit of determination of the laboratory for BOD and two-thirds of the initial oxygen concentration.

NOTE 2: Care should be taken that representative samples are collected.

8 Calculation and expression of results

Calculate the biochemical oxygen demand after n days (BOD_n), expressed in milligrams per litre of oxygen, using the equation

$$BOD_n = (c_1 - c_2) \qquad (6)$$

where:

c_1 is the dissolved oxygen concentration of the test sample at time zero, in milligrams per litre;

c_2 is the dissolved oxygen concentration of this same test sample after n days, in milligrams per litre.

The results shall be reported to two significant figures, e.g. 4,5 mg/l of oxygen.

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9 Trueness and Precision

The standard deviation of the BOD_n-determination was determined by an interlaboratory comparison in 1992 where 76 laboratories from nine countries performed 2 to 4 analyses on two stabilized fresh, surface water samples and two locally collected fresh, surface water samples.

Table 1: Interlaboratory comparison 46:1992 - Results

	Sample type		Median	Repeatability Standard deviation	Relative standard deviation within laboratories	Reproducibility Standard deviation	Relative standard deviation between laboratories	No. of laboratories included in calculations	Outlier laboratories
			mg/l of oxygen	mg/l of oxygen	%	mg/l of oxygen	%		
BOD_5	Stabilized fresh water	A	2,15	0,10	4,3	0,53	24	72	2
	Stabilized fresh water	B	4,87	0,13	2,7	0,85	18	72	3
	Natural fresh water	C	1,56	0,12	7,4	–	–	24	3
	Natural fresh water	C_o	0,68	0,13	18	0,26	36	16	1
	Natural fresh water	C_1	1,29	0,13	9,4	0,34	26	28	1
	Natural fresh water	D	2,06	0,15	5,0	–	–	24	1
	Natural fresh water	D_s	4,69	0,22	4,8	0,30	6,4	16	0
	Natural fresh water	D_1	3,03	0,15	4,7	0,31	10	28	0
BOD_{2+5}	Stabilized fresh water	A	2,12	0,13	5,9	0,37	17	71	2
	Stabilized fresh water	B	4,92	0,17	3,6	0,85	18	71	3
	Natural fresh water	C	1,29	0,12	7,4	–	–	24	2
	Natural fresh water	C_o	0,62	0,10	17	0,21	36	16	1
	Natural fresh water	C_1	1,28	0,11	9,1	0,27	21	28	0
	Natural fresh water	D	1,90	0,10	3,9	–	–	24	3
	Natural fresh water	D_s	4,68	0,15	3,1	0,39	8,4	16	1
	Natural fresh water	D_1	3,22	0,13	3,9	0,38	12	28	1
BOD_7	Stabilized fresh water	A	2,57	0,11	4,3	0,40	15	71	6
	Stabilized fresh water	B	5,82	0,15	2,7	0,94	17	71	4
	Natural fresh water	C	2,02	0,13	7,5	–	–	24	4
	Natural fresh water	C_o	0,90	0,08	9,8	0,26	30	16	4
	Natural fresh water	C_1	1,50	0,14	8,9	0,38	25	28	2
	Natural fresh water	D	2,67	0,17	4,9	–	–	24	2
	Natural fresh water	D_s	5,51	0,29	5,3	0,42	7,7	16	0
	Natural fresh water	D_1	4,74	0,16	3,4	0,44	9,3	28	0

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In this interlaboratory comparison, a repeatability standard deviation of (0,10 to 0,29) mg/l of oxygen and a reproducibility standard deviation of (0,26 to 0,94) mg/l of oxygen was found.

It is possible to establish factors for conversion between BOD_5 and BOD_7 data within a single type of water.

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The value of conversion factors can be obtained for each water type from parallel analyses of BOD_5 and BOD_7 measurements of the same samples. If a factor is not available the correlation between BOD_5 and BOD_7 can be estimated from the above mentioned European interlaboratory comparison. The results are shown in table 2.

Table 2: Interlaboratory Comparison 46:1992 - Comparison of BOD_5 and BOD_7 determinations

Sample type		BOD_5 mg/l of oxygen Median	BOD_7 mg/l of oxygen Median	Significant Difference*)	Number of laboratories included in calculations	BOD_7/BOD_5
Stabilized fresh water	A	2,15	2,57	Yes	71	1,20
	B	4,87	5,82	Yes	71	1,20
Natural fresh water	C_s	0,68	0,90	Yes	15	1,32
	C_j	1,29	1,50	Yes	28	1,16
Natural fresh water	D_s	4,69	5,51	Yes	16	1,28
	D_j	3,03	4,74	Yes	28	1,56
*) significance level $\alpha = 0,05$						

10 Test report

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The test report shall include the following information:

a) a reference to this European Standard;

b) the number of days of incubation (n);

c) the results in milligrams per litre of oxygen (reported as described in clause 8);

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d) for results below the working range, a documentation for an adequate limit of determination;

e) any special details which may have been noted during the test;

f) details of any operations not specified in this European Standard, or regarded as optional, such as aeration (see 7.1), alternative incubation (BOD_{2+5}) (in accordance with Annex A) and modifications such as freezing and homogenization (in accordance with Annex B).