

ANALYTICAL EQUIPMENT FOR



ECOLOGY AND POWER INDUSTRY

MARK-302T DISSOLVED OXYGEN METER

Operation Manual



АЯ 74

Nizhny Novgorod
2010

VZOR will appreciate any suggestions and comments aimed at product quality improvement.

If you have any trouble with the instrument performance, please contact us in writing or by phone.

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1 DESCRIPTION AND OPERATION

1.1 Purpose

1.1.1 Product name and designation

MARK-302T dissolved oxygen analyzer

TU 4215-022-39232169-2008.

1.1.2 The analyzer has been designed to measure the dissolved oxygen concentration and water temperature.

1.1.3 The analyzer's scope of application is high-sensitivity measurement of the mass concentration of dissolved oxygen (in the microgram range). It is used primarily at heat power facilities for deaerated water monitoring.

1.1.4 The analyzer may also be used to measure the mass concentration of water-dissolved oxygen and temperature of surface, waste and potable water, and may be employed by fish farms; chemical, biotechnological and food industries; educational institutions and ecology branches.

1.1.5 The type of analyzer:

- amperometric;
- with external poling voltage;
- with one sensitive element;
- with a digital LCD display;
- with automatic temperature compensation;
- with flow-type/ dip sensor;
- with automatic calibration when the sensor is placed in the oxygen environment (air) at temperatures varying between +15 °C and +35 °C;
- with automatic adjustment for atmospheric pressure during calibration.

1.2 Basic parameters

1.2.1 By resistance to climatic effects, the analyzer version group is B4 in accordance with GOST 12997-84.

1.2.2 By resistance to mechanical effects, the analyzer version is L1 in accordance with GOST 12997-84.

1.2.3 The converter unit degree of protection ensured by the casing is IP30 in accordance with GOST 14254-96.

1.2.4 By resistance to atmospheric pressure, the analyzer version group is P1 in accordance with GOST 12997-84 (atmospheric pressure between 84 and 106.7 kPa).

1.2.5 Analyzed water parameters:

- temperature, °C 0 to +50;
- pressure, MPa, max 0.05;
- salt content, g/dm³ 0 to 40;
- pH 4 to 12;
- rate of water flow via flowing vat, cm³/min 400 to 800;
- rate of water flow relative to sensor membrane, cm/s 5.

1.2.6 Permissible concentrations of unmeasured components:

- dissolved ammonia, mg/dm³, max 40.0;
- dissolved phenol, mg/dm³, max 0.2.

1.2.7 Operating conditions:

- ambient air temperature, °C +1 to +50;
- relative air humidity at 35 °C and lower temperatures without moisture condensation, %, max 80;
- atmospheric pressure, kPa (mm hg) 84.0 to 106.7
(630 to 800).

1.2.8 The analyzer is calibrated by air of 100 % humidity.

Calibration temperature range, °C +15 to +35.

1.2.9 The analyzer is powered from a 2,2-3,4 V DC self-contained power supply (two AA-type alkaline batteries).

1.2.10 Power consumption (at a rated voltage of 2,8 V), mW, max 10.

1.2.11 The analyzer retains its characteristics within the ranges set in the analyzer specifications after replacement of the sensor's spare parts, batteries and calibration.

1.2.12 The overall dimensions and weights of analyzer components are summarized in Table 1.1 below.

Table 1.1

Name and designation of component versions	Overall dimensions, mm, max	Weight, kg, max
BP29.01.000 converter unit	84×160×38	0,30
BP29.02.000 oxygen sensor (without cable)	∅16×115	0,12
BP29.03.000 flowing vat	18×40×121	0,10

1.2.13 GOST 12997-84 conditions for carriage in shipping containers:

- temperature, °C – 20 to +50;
- relative air humidity at 35 °C, % 95±3;
- 5-35 Hz sinusoidal vibration with a 0.35 mm shift amplitude in the direction marked "Top. Do not turn over" on the package.

1.3 Specifications

1.3.1 DOC (dissolved oxygen concentration) measuring range at an analyte temperature of 20 °C, mg/dm³ 0 to 10,00.

The upper limits of DOC measuring range, depending on the analyte temperature, are shown in Table 1.2.

Table 1.2

t, °C	0	5	10	15	20	25	30	35	40	45	50
DOC, mg/dm ³	17,45	15,29	13,48	12,10	10,00	9,85	8,98	8,30	7,69	7,12	6,59

1.3.2 Limits of the analyzer allowable basic absolute error in DOC measurement at an analyte temperature of (20,0±0,2) °C and ambient temperature of (20±5) °C, mg/dm³ ±(0,003+0,04C), where C, here and hereafter, is a measured DOC value in mg/dm³.

1.3.3 Limits of the analyzer allowable additional absolute error in DOC measurement due to a change in the ambient temperature, by each ±5 °C from the normal temperature (20,0±0,2) °C, within the operating temperature range from 0 to +50 °C, mg/dm³ ±0,012C.

1.3.4 Limits of the analyzer allowable additional absolute error in DOC measurement due to a change in the ambient temperature by each ±10 °C from the normal temperature (20±5) °C, within the operating temperature range from +1 to +50 °C, mg/dm³ ±(0,001+0,002C).

1.3.5 Limits of the analyzer allowable absolute error in DOC measurement at an analyte temperature coinciding with the calibration temperature which varies between +15 °C and +35 °C, at an ambient temperature of (20±5) °C, mg/dm³ ±(0,003+0,04C).

1.3.6 Analyte temperature measuring range, °C 0 to +50.

1.3.7 Limits of the analyzer allowable basic absolute error in analyte temperature measurement at an ambient temperature of (20±5) °C, °C ±0,3.

1.3.8 Limits of the analyzer allowable basic absolute error in analyte temperature measurement due to a change in the ambient temperature by each ±10 °C from the normal temperature (20±5) °C, within the operating temperature range from +1 to +50 °C, °C ±0,1.

1.3.9 Limit of the permissible value of the analyzer readout setting time $t_{0,9}$ in DOC measurement, min 2.

1.3.10 Limit of the permissible value of the analyzer readout setting time t_y in DOC measurement, min 30.

1.3.11 Limit of the permissible value of the analyzer readout setting time $t_{0,9}$ in analyte temperature measurement, min 1.

1.3.12 Limit of the permissible value of the analyzer readout setting time t_y in analyte temperature measurement, min 3.

1.3.13 Analyzer oscillatory readings in DOC measurement for 8 h, mg/dm³,
 max ±(0,0015+0,02C).

1.4 Product components

The analyzer comprises:

- converter unit;
- oxygen sensor with a 2 m connecting cable;
- flowing vat.

1.5 Description and operation

1.5.1 General analyzer data

The MARK-302T dissolved oxygen analyzer is a small-size microprocessor device designed to measure the mass concentration of water-dissolved oxygen (DOC) and the analyte temperature.

The DOC values measured in mg/dm³ or temperature measured in Celsius degrees (depending on the measuring mode) are displayed on the readout device – a digital LCD display (“the display”). The minimum value of the least significant digit for DOC measurement is 0,001 mg/dm³. The least significant digit value for temperature measurement is 0,1 °C.

The analyzer is calibrated by atmospheric air of 100 % humidity with automatic adjustment for atmospheric pressure during calibration.

Adjustment for atmospheric pressure during the analyzer calibration by atmospheric air is provided by a built-in atmospheric pressure sensor.

1.5.2 Analyzer operating principle

The analyzer uses an amperometric sensor operating as a closed-type polarographic cell. The electrodes are submerged in an internal electrolyte solution which is separated from the analyte by a membrane penetrable for oxygen but impenetrable for liquid and water vapor. From the analyte, oxygen diffuses through the membrane into a thin layer of electrolyte between the electrodes and the membrane and undergoes an electrochemical reaction on the surface of the cathode which is polarized by external voltage applied across the electrodes. In the process, the sensor generates a DC signal which is at a fixed temperature proportional to

DOC in the analyte.

The analyzer features a temperature sensor (platinum thermal resistor) to measure temperature and to automatically compensate for the temperature dependence of the oxygen sensor signal. The temperature sensor signal is supplied to the ADC input.

The ADC converts signals of the oxygen sensor and the temperature sensor into codes incoming the microcontroller.

The microcontroller processes the codes received and shows the information on the digital LCD display.

1.5.3 Analyzer design

The analyzer is shown in Fig.1.1a.

Converter unit 1 has a sealed plastic housing and is designed to convert signals from oxygen sensor 2 and display measurement data.

The front panel of the converter unit features:

- indicator screen 3 designed to display a measured DOC value or temperature (depending on the chosen measuring mode), battery charge and current time (in off state);
- button 4.

The back panel of the converter unit features a battery compartment cover.

Located on top of the converter unit is sealed cable entry 5 for oxygen sensor 2.

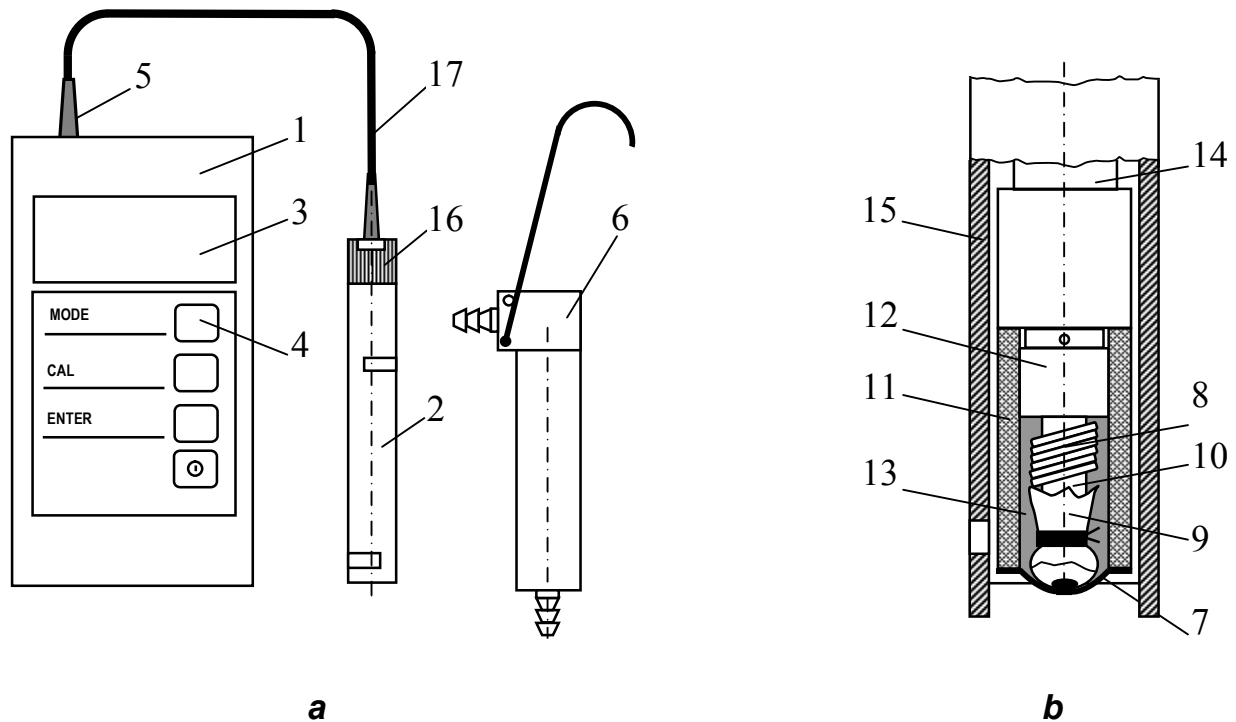


Fig. 1.1

Flow measurements use flowing vat 6.

Fig.1.1b shows the oxygen sensor design.


The sensor's basic functional elements are platinum cathode 7 and silver anode 8. Secured to cathode 7 with caprone thread 9 is teflon film 10. The membrane and rubber bushing form membrane assembly 11 fitted over bushing 12 and filled with electrolyte 13. The temperature sensor is enclosed in case 14. Protective bushing 15 covers the sensor electrode portion and is screwed into cable bushing 16 (Fig.1.1a).

Cable 17 connects the sensor electrode assembly to the converter unit.

Made of a corrosion resistant alloy, flowing vat 6 is a threaded cylinder with supply and drain connections for controlled water. To install the oxygen sensor in the flowing vat, replace protection bushing 15 with flowing vat 6.

1.5.4 Functions of buttons on the converter unit front panel

The analyzer front panel features the following buttons as shown in Fig.1.1a:

- the  button for analyzer power on/off. When the analyzer is energized, the display shows the measured DOC or temperature value;
- the **MODE** button enables the DOC or temperature measuring mode, with the display showing the measured DOC or temperature value and **mg/dm³** or **°C** character, respectively, coming on in the right half of the indicator;
- the **CAL** button selects the analyzer calibration mode. When pressed sequentially, the button causes the characters **c0** (analyzer reset) and **c1** (atmospheric air calibration) to light up on the display;
- the **ENTER** button confirms the selected calibration mode and completes calibration.

1.6 Measuring instruments, tools and accessories

1.6.1 The following tools and accessories, which are not included in the delivery set, will be additionally required for maintenance of the analyzer:

- 2 mm cross screwdriver;
- B-1-250 beaker;
- KH-100-19/26 flask;
- hydroquinone, chemically pure;
- sodium or potassium hydroxide, chemically pure.

2 INTENDED USE

2.1 Operating limitations

2.1.1 2.1.1 The MARK-302T analyzer version is primarily used to measure DOC in deaerated water.

2.1.2 2.1.2 Para.1.2.5 provides permissible concentrations for a number of components which affect measuring results.

2.1.3 2.1.3 The analyzer should be positioned so as to prevent water from getting on the converter unit.

2.1.4 2.1.4 When using the analyzer, avoid hitting or dropping the oxygen sensor to keep its glass components intact.

2.2 Safety

2.2.1 The analyzer should only be operated by personnel who are familiar with this manual and chemical handling safety rules in accordance with GOST 12.1.007-76 and GOST 12.4.021-75.

2.2.2 The analyzer meets the requirements of Safety Class III to GOST R 52319-2005. The rated supply voltage varies between 2,2 and 3,4 V. No protective earthing is required.

2.2.3 By electromagnetic compatibility, the analyzer meets the requirements of GOST R 51522-99 for Class B equipment.

2.3 Pre-starting procedures

On receipt of the analyzer, unpack it, check the set for completeness and make sure that the packed components are intact.

If kept in cold conditions before delivery, the analyzer should be held at room temperature for at least one hour before setting-up procedures.

2.3.1 Power supply connection

To connect the power supply, remove the battery compartment cover located on the rear panel of the converter unit. Install two AA-type alkaline batteries observing the polarity marking inside the battery compartment. Replace the battery compartment cover.

With a power supply installed in the battery compartment, the analyzer can indicate time, when turned off. The dot between hours and minutes flickers at an interval of 1 second.

Time indication may be switched off and on again by pressing the **MODE** button, with the analyzer off.

Proceed as follows to correct time:

- press the **CAL** button, minutes will start blinking on the display;
- use the **MODE** and **ENTER** buttons to set the minute value;
- press the **CAL** button, hours will start blinking on the display;
- use the **MODE** and **ENTER** buttons to set the hour value;
- press the **CAL** button to complete time setting, the analyzer goes into the time indication mode.

Switch on the analyzer and check if the display shows DOC or temperature readings in mg/dm³ or °C respectively.

IMPORTANT: ALWAYS OBSERVE polarity when installing batteries. Failure to meet this condition may damage the analyzer!

When the display shows the  sign, replace AA-type alkaline batteries.

2.3.2 Oxygen sensor setting-up procedures

Since the oxygen sensor from the analyzer set is delivered in the “dry” state, it should be filled with electrolyte included with the delivery set, as described in para.2.6.3, and submerged in distilled water for a minimum of 8 hours.

Make sure two AA-type alkaline batteries are installed in the converter unit. Regardless of whether the analyzer is on or off, the sensor will receive polarizing voltage needed to build up an electrode system.

2.3.3 Analyzer functional check

The analyzer functional check includes:

- analyzer precalibration by atmospheric air oxygen;
- check of readings in the null solution.

It is recommended that the analyzer functional check be performed:

- after filling the sensor with electrolyte on delivery of the analyzer;
- after replacement of the membrane assembly or teflon film;
- if there are any doubts as to the analyzer's serviceability.

2.3.3.1 Analyzer precalibration

Take the sensor out of the vessel with water and place it horizontally (put on the table).

Press the **MODE** button to enable the DOC measuring mode in mg/dm^3 . The analyzer display will show a figure measured in mg/dm^3 .

Keep the sensor in the air for 5 min.

Press the **CAL** button twice. The analyzer display will show the **c1** symbol showing the entry into the outside air calibration mode.

Press the **ENTER** button. The analyzer display will show DOC readings corresponding to the table of 100 % humidity air oxygen solubility for analyzer-measured temperatures, with allowance for the atmospheric pressure at the time of calibration, for example, **c8.38 mg/dm^3** .

Wait at least **8 s** before pressing the **ENTER** button once again. The **donE** caption will appear shortly on the screen and the **c** symbol will go out. The analyzer will go into the measuring mode, which means that precalibration is completed.

2.3.3.2 Reading check in the null solution

Prepare an oxygen-free (*null*) solution, and for this purpose:

- prepare an alkali solution (KOH or NaOH), concentration 5 g/dm^3 ;
- pour it into a $0,3\text{-}0,5 \text{ dm}^3$ vessel so that its level is 50 to 60 mm;
- add 0,3 to 0,5 g of hydroquinone and stir.

Uptime in air tight vessel is max 1 month.

Submerge the sensor, with its membrane down, in the solution and move it to slightly agitate the solution in order to prevent air bubbles from amassing on the membrane. The displayed analyzer reading should decrease slowly.

Take the analyzer readings in 30 min.

They should vary within $\pm 3 \text{ mkg/dm}^3$.

With the above procedure successfully completed, the analyzer is ready for normal service. The analyzer calibration by outside air shall then be performed as per para.2.3.4.

If the readings do not decrease to the specified level, a sensor cycling operation should be carried out as per para.2.3.3.3.

2.3.3.3 Sensor cycling

Proceed as follows to carry out the cycling operation:

- switch on the analyzer;
- prepare a null solution as per para.2.3.3.2;
- submerge the sensor, with its membrane down, in the solution and move it to slightly agitate the solution in order to prevent air bubbles from amassing on the membrane;
 - hold the sensor in the null solution for 5 min, then take it out, shake off the solution drops from the membrane and hold it in the air for 5 min;
 - repeat the null solution - air cycle 3 to 4 times;
 - submerge the sensor in null solution again;
 - take the analyzer reading in 30 min; it should be within ± 3 mkg/dm³.

If the above steps fail to cause the analyzer readings in the null solution to decrease to the desired values, this suggests either bad quality of the null solution (bad reagents) or a fault in the analyzer (see section 2.6 Troubleshooting).

Calibration of the analyzer should then be performed subject to para.2.3.4 or 2.3.5.

If any error character (**E3**, **E4**, **E5**, **E6**, **E7**, **E8**, **E9**) is displayed during the analyzer functional check, also refer to section 2.6.

2.3.4 Analyzer calibration by outside air

Outside air calibration of the analyzer should be performed:

- when the device is new;
- once a shift (8 hr);
- after replacement of electrolyte, membrane and teflon film.

Calibration of the analyzer is performed in the outside air at temperatures between +15 °C and +35 °C and a 100 % relative humidity. It is more convenient to perform calibration at room temperature.

Prior to calibration, the analyzer, with AA-type alkaline batteries installed, should be held at room temperature for at least 1 hour. The analyzer may be left

switched on. Submerge the sensor fully in distilled water of room temperature for a minimum of 10 min.

Rinse the sensor in distilled water, shake off water drops from the sensor membrane and put the sensor in the cone flask with a water layer 3 to 5 mm, as shown in Fig.2.1. Position the flask at an angle of 30 to 45° to the horizontal for the remaining water to flow off the membrane.

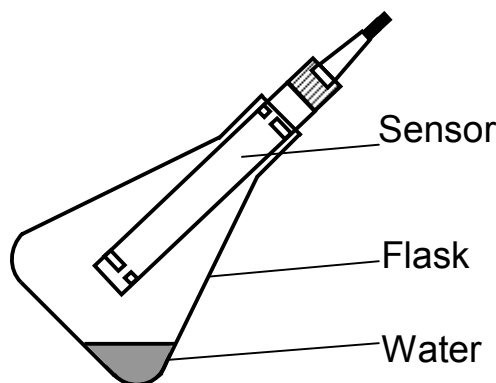


Fig. 2.1 – Position of the sensor inside the flask for analyzer calibration

Wait for 10 min before performing the outside air calibration steps which follow.

1 Press the **CAL** button twice. The **c1** character will appear on the display, showing the entry into the outside air calibration mode.

2 Press the **ENTER** button. The analyzer will display DOC readings corresponding to the table of 100 % humidity air oxygen solubility for analyzer-measured temperature, with allowance for the atmospheric pressure at the time of calibration, e.g. **c8.38 mg/dm³**.

3 Wait at least **8 s** before pressing **ENTER** once again. The **donE** caption will briefly appear on the display and the analyzer will go into the measuring mode. It means the outside air calibration mode is completed and the analyzer is calibrated.

Once calibrated by outside air, the analyzer is ready for use.

Note: Outside air calibration of the analyzer may be cancelled before Step 3 by pressing **MODE**. The analyzer will go into the DOC measuring mode, retaining the values of previous calibration coefficients.

2.3.5 Analyzer zero calibration

Analyzer zero calibration allows the sensor residual zero current to be compensated within narrow limits (–3,0 to +3,0 mkg/dm³).

Proceeds as follows prior to carrying out this operation:

- switch on the analyzer;
- prepare a fresh null solution as per para.2.3.3.2;
- enable the DOC measuring mode in mg/dm^3 ;
- perform cycling operations as per para.2.3.3.3;
- hold the sensor in the air for 5 min, submerge the sensor, with its membrane down, in the null solution and move it to slightly agitate the solution in order to prevent air bubbles from amassing on the membrane;
- hold the sensor in the null solution for at least 40 min.

Perform the following steps to set the analyzer zero.

- 1** Press **CAL**. The analyzer display will show the **c0** caption.
- 2** Press **ENTER**. The analyzer display will show DOC Z_0 readings in the null solution without allowance for zero adjustment, e.g. **c.002 mg/dm³**.
- 3** Wait at least **8 s** before pressing **ENTER** once again. The **donE** caption will briefly appear on the display and the analyzer will go into the measuring mode. The analyzer display will indicate a DOC value in the null solution after analyzer zero setting:

- | | | |
|-------------------|----|--|
| – 0,000; | if | $-0,003 \text{ mg/dm}^3 \leq Z_0 \leq 0,003 \text{ mg/dm}^3$; |
| – $Z_0 - 0,003$; | if | $Z_0 > 0,003 \text{ mg/dm}^3$; |
| – $Z_0 + 0,003$; | if | $Z_0 < -0,003 \text{ mg/dm}^3$. |

Note: Analyzer zero setting may be cancelled before Step 3, by pressing **MODE**. The analyzer will go into the DOC measuring mode, retaining the values of previous calibration coefficients.

2.4 Measurement procedure

2.4.1 Preparation for measurement using a flowing vat

The premeasurement procedure as per Fig.2.2 includes the following steps:

- remove the hose from the flowing vat outlet connection;
- remove the protective bushing from the cable bushing by unscrewing it;
- screw in the flowing vat in place of the cable bushing.

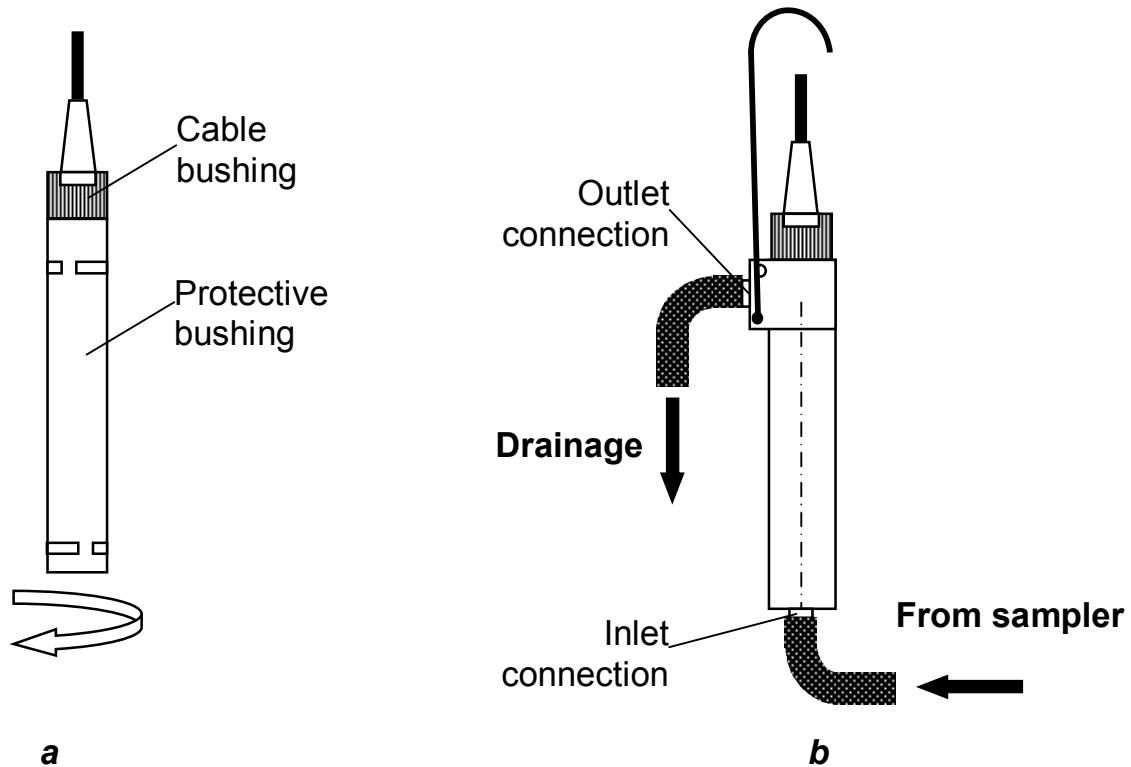


Fig. 2.2

2.4.1.1 Measurement using a flowing vat

Connect the inlet connection of the flowing vat (with the sensor installed) to the controlled water line with a flexible hose. Supply controlled water to the flowing vat and position the vessel with sensor so that the sensor is nearly vertical, its membrane down. Allow water to freely flow through the vessel for at least 10 minutes, until there are no air bubbles in the water flow. Nor should there be any air bubbles on the sensor membrane. To remove bubbles from the membrane, shake the vessel with sensor carefully.

Bubbles amassing in the hose bends, on the sensor membrane or in the water line knee may substantially invalidate the results. One of the signs showing the presence of air bubbles is that the analyzer oxygen readings will not settle, while dropping slowly and continuously. Such a process caused by the air oxygen being washed out from air bubbles may last 1-2 hours.

To eliminate air bubbles in the sampler line, it is recommended:

- to sharply increase (by 10-20 s) the water flow through the flowing vat;
- reduce the water flow to the normal level (400 to 800 cm³/min).

Switch on the analyzer and take the display readings.

Negative oxygen readings at sampler measurements suggest that the analyzed water contains some electroactive impurities.

Measurements may dispense with the flowing vat, if the sensor is placed in a suitable vessel ensuring a controlled water flow of 5 cm/s in the sensor membrane area. To protect the membrane from damage, it is recommended to screw in the protective bushing.

Note: Subject to para.1.3.11, the MARK-302T analyzer readout setting time limit t_y , when measuring DOC, is 30 min, i.e. after 30 min the analyzer readings in a fresh null solution should be up to 0,003 mg/dm³.

The real readout setting time for newly released analyzers varies between 1 and 3 min.

When in use for a certain period, the analyzer may have an extended readout setting time.

To determine the readout setting time of a specific analyzer, prepare a fresh null solution, submerge the sensor in it, slightly agitating the solution with the sensor, and record the time when the readings of 0,003 mg/dm³ are obtained. It is recommended to perform this operation once a month.

The 0,003 mg/dm³ reading recorded time may be used for measurement, i.e. take reading when this time expires.

IMPORTANT: while operating the analyzer:

- keep the sensor membrane wet. The sensor should be kept in water during intervals between measurements. There is a good reason to store the sensor installed in the flowing vat filled with controlled water. To prevent water leakage, the hoses of inlet and outlet connections may be connected with a short pipe;
- transport the oxygen sensor in the flowing vat filled with water, at positive ambient temperatures;
- when brought into a warm room from the cold air, prior to enabling, hold the analyzer at room temperature for at least 1 hour in order for condensed moisture to evaporate.

2.4.2 Water temperature measurement

The temperature measurement mode **t °C** is enabled by pressing the **MODE** button.

Allow for the analyzer readings to be set and record them as a measuring result.

2.4.3 Calculation of dissolved oxygen concentration by analyzer readings, with allowance for the salt content

When measuring DOC in salty water, use the correction factor α , by which the analyzer readings should be multiplied. The α value is defined with the formula:

$$\alpha = 1 - C_{salt} \cdot \varepsilon,$$

where C_{salt} – salt content, g/dm³;
 ε – factor listed in Table 2.1.

Table 2.1. – Correction factors

t °C	ε	t °C	ε	t °C	ε	t °C	ε	t °C	ε
0,0	0,0063	11,0	0,0057	21,0	0,0052	31,0	0,0048	41,0	0,0043
1,0	0,0063	12,0	0,0057	22,0	0,0052	32,0	0,0047	42,0	0,0042
2,0	0,0062	13,0	0,0057	23,0	0,0051	33,0	0,0047	43,0	0,0042
3,0	0,0062	14,0	0,0055	24,0	0,0050	34,0	0,0046	44,0	0,0041
4,0	0,0060	15,0	0,0055	25,0	0,0050	35,0	0,0046	45,0	0,0041
5,0	0,0060	16,0	0,0055	26,0	0,0049	36,0	0,0045	46,0	0,0040
6,0	0,0060	17,0	0,0054	27,0	0,0049	37,0	0,0045	47,0	0,0040
7,0	0,0060	18,0	0,0054	28,0	0,0049	38,0	0,0044	48,0	0,0039
8,0	0,0058	19,0	0,0053	29,0	0,0048	39,0	0,0044	49,0	0,0039
9,0	0,0058	20,0	0,0053	30,0	0,0048	40,0	0,0043	50,0	0,0038
10,0	0,0058								

Exemplary calculation of correction factor α :

Let $C_{salt} = 10$ g/dm³, $t = 20$ °C,
 thus $\varepsilon = 0,0053$,
 then $\alpha = 1 - 10 \cdot 0,0053 = 0,947$.

Note: This method for salt content correction is based on the International Standard ISO 5814 Water Quality – Determination Of Dissolved Oxygen – Electrochemical Probe Method.

2.5 Functional check

A serviceable analyzer should meet the following requirements:

– with the sensor placed in the null solution, the analyzer readings stay within ± 3 mkg/dm³;

– when outside air calibration is accomplished, (para.2.3.4) neither **E3** nor **E4** is displayed on the screen and readings C_{cal} , mg/dm³, are set with an accuracy ± 1 % of the calculated value defined with the formula:

$$C_{cal} = Co_2(t) \cdot \frac{P_{atm}}{101.325},$$

where $Co_2(t)$, mg/dm³ – solubility of 100 % humidity air oxygen in distilled water, at temperature t , °C, and normal atmospheric pressure of 101.325 kPa as per Table A.1;

P_{atm} – atmospheric pressure as of the time of calibration, kPa.

2.6 Troubleshooting

2.6.1 The typical faults of the analyzer and remedial methods are summarized in Table 2.2.

In case of troubles set out in Table 2.2, proceed as recommended in the Remedy column, in accordance with the clauses given below and Fig.1.1 and 2.4.

Table 2.2

Trouble, symptoms	Probable cause	Remedy
1 With power on, no readings are displayed	Poor battery contact	Open the battery compartment and clean contacts
	Supply voltage below acceptable level	Para.2.3.1 Replace batteries
2 With power on, all or random segments and characters illuminate on the screen	Dead batteries	Para.2.3.1. Replace batteries
3 When the zero point of measuring range is checked, analyzer readings go beyond $\pm 0,003$ mg/dm ³	Ruptured or punctured membrane, sensor seal failure	Para.2.6.3, 2.6.4. Replace the membrane and electrolyte
	Moisture inside the converter unit	Dry the converter unit for 3-4 days
	Overstretched membrane	Para.2.6.4. Replace the membrane assembly
	Bad null solution	Replace the null solution
	Broken (cracked) glass holder of sensor electrodes	Factory repair

Table 2.2 (Continue)


Trouble, symptoms	Probable cause	Remedy
4 When the analyzer is calibrated by outside air, E3 (sensor current below rating) is displayed.	Electrolyte has leaked out	Para.2.6.3. Fill in electrolyte
	Contaminated membrane	Para.2.6.2. Clean the membrane
	Dry membrane	Keep the membrane in water for 2-3 days without dismantling the sensor
	Defective membrane	Para.2.6.4. Replace the membrane assembly
	Analyzer sensor off the outside air	Position the sensor in air
5 Electrolyte leaking out fast	Ruptured membrane	Para.2.6.4. Replace the membrane assembly
6.1 Sharp change in and increased instability of analyzer readings. 6.2 During analyzer calibration by outside air, E4 (excess sensor current) is displayed on the screen.	Ruptured membrane	Para.2.6.4. Replace the membrane assembly
	Contaminated electrolyte	Para.2.6.3. Replace electrolyte
	Moisture inside the converter unit	Dry the converter unit for 3-4 days
	Ruptured teflon film	Para.2.6.4. Replace teflon film
	Analyzer sensor off the outside air	Position the sensor in air
7 Extended time of response to oxygen concentration variations	Contaminated membrane	Para.2.6.2. Clean the membrane
	Contaminated platinum electrode	Para.2.6.5. Clean the platinum electrode
8 During measurements, E5 (excess measured sensor current) is displayed on the screen. Analyzer does not respond to pressed buttons, except button 	Ruptured membrane	Para.2.6.4. Replace the membrane assembly
	Contaminated electrolyte	Para.2.6.3. Replace electrolyte
	Moisture inside the converter unit	Dry the converter unit for 3-4 days
	Ruptured teflon film	Para.2.6.4. Replace teflon film
	Broken down analyzer	Factory repair

Table 2.2 (Continue)

Trouble, symptoms	Probable cause	Remedy
9 During measurements, indication of exceeded display digit capacity appears on the screen: “E6 mg/dm ³ ” – readings less than 199,9 mg/dm ³ ; “E7 mg/dm ³ ” – readings exceeding 199,9 mg/dm ³ .	Operator’s errors in analyzer calibrations	Para.2.6.6. Perform operations to set initial analyzer parameters
	Broken down analyzer	Factory repair
10 During measurements, E8 is displayed on the screen.	Faulty temperature measuring channel (heat probe breakage)	Factory repair
11 E9 is displayed on the screen.	Writing error in EEPROM memory	Factory repair
12 Sharp change in and increased instability of analyzer readings during flowing vat measurements.	High rate of flow through the flowing vat	Set the rate of flow through the flowing vat at 400 to 800 cm ³ /min

2.6.2 Membrane cleaning

The sensor membrane may be cleaned with a wad of alcohol-soaked cotton.

The sensor membrane may also be submerged in a weak solution (2 %) of sulfuric acid for about 1 hour, and then wash it in running water.

2.6.3 Filling the sensor with electrolyte, electrolyte replacement

As it is supplied in a dry state (without electrolyte), the sensor needs to be filled with electrolyte after its receipt from the manufacturer.

Unscrew and remove the protective bushing from the sensor as per Fig.2.3.

Remove the membrane assembly from the bushing. Draw electrolyte from SPK into a syringe. Take the membrane assembly and, holding it with the membrane down, inject electrolyte to fill two thirds of the volume, taking care to avoid damaging the membrane. While continuing to keep vertical the membrane assembly filled with electrolyte, set it on the bushing against the stop. Screw in the protective bushing.

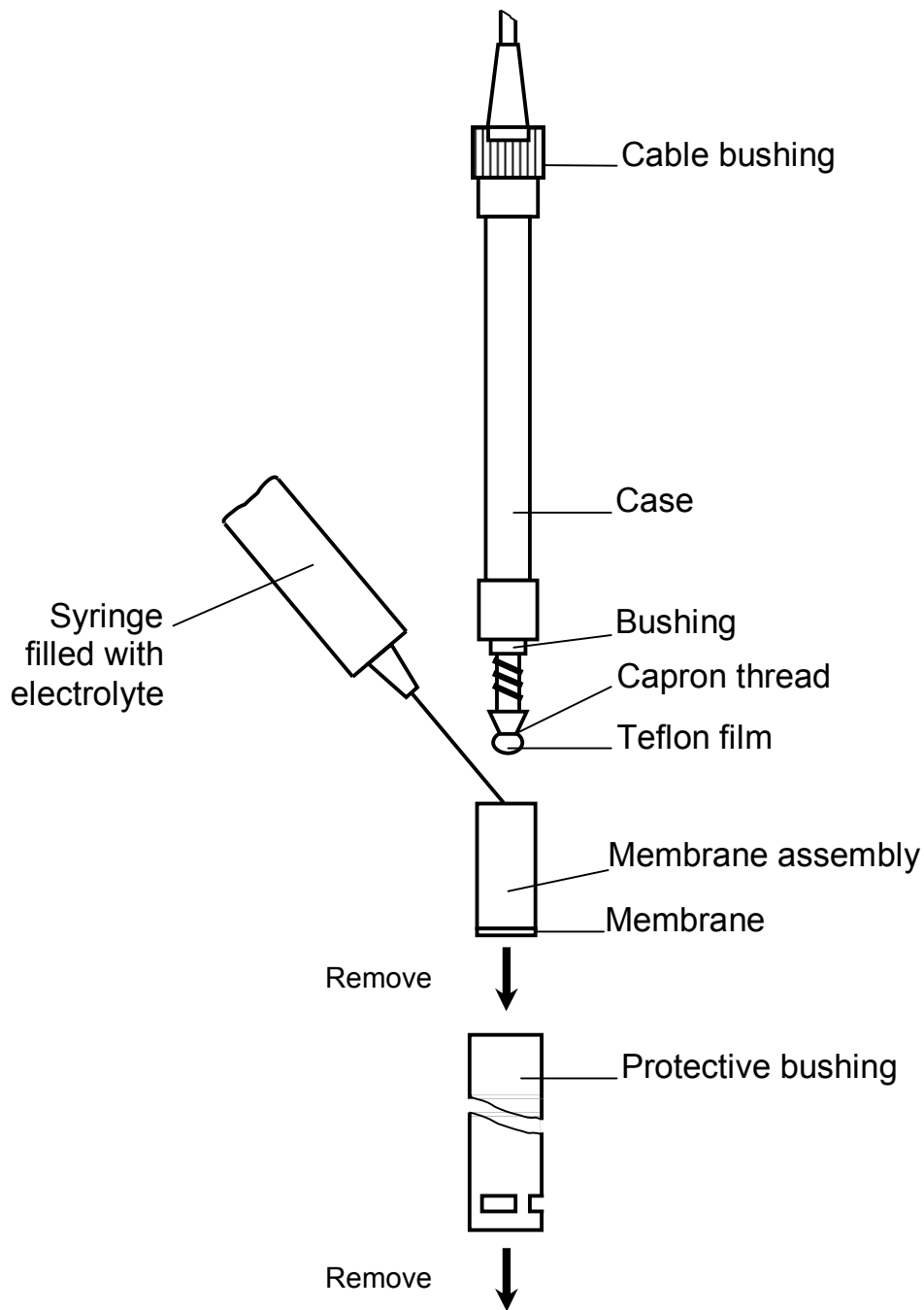


Fig. 2.3 – Dismantling the sensor for filling and replacement of electrolyte, replacement of teflon film and membrane assembly

IMPORTANT: The membrane should be stretched and pressed firmly against the sensor platinum cathode. The membrane **SHALL NEVER** separate from the cathode!

After a certain period of service, the sensor may have the volume of electrolyte reducing due to leakage through pinholes or ruptures in the membrane, which necessitates electrolyte replacement.

Remove the membrane assembly from bushing 6, drain the remaining electrolyte and wash the assembly with distilled water. Fill the membrane assembly with new electrolyte.

Electrolyte composition: KCl, chemically pure – 14 g; KOH, chemically pure – 0.2 g; versene – 0.15 g; distilled water – up to 0.1 dm³. Filter the solution.

2.6.4 Replacement of the membrane assembly and teflon film

2.6.4.1 Replacement of the membrane assembly may be necessary in case of mechanical damage (cracks, ruptures) or overstretching of the membrane. A faulty membrane manifests itself in analyzer reading instability, high analyzer readings in the null solution and longer time of response during DOC measurement.

Unscrew and remove the protective bushing from the sensor as per Fig.2.3. Remove the membrane assembly from the inner case and drain the electrolyte.

Check the teflon film for damage.

The film should be firmly pressed against the cathode, in a wrinkle-free manner. Damaged film should be replaced.

Once the teflon film is removed, check the sensor electrode to see if they look as follows:

- platinum cathode 1 (Fig.1.1b) sealed in a glass tube should be clean;
- silver anode 2, coiled over the tube should be gray.

If necessary, clean the electrodes with a wad of alcohol-soaked cotton.

IMPORTANT: NEVER use any abrasive for cleaning electrodes!

2.6.4.2 Damaged teflon film should be replaced with new one from spare parts kit. To do this, place it on the cathode, press the film edges firmly against the side surface of the glass tube and, holding the edges by hand, wind around 5 to 6 laps of caprone thread making 2 to 3 knots. Cut off with scissors surpluses of teflon film not closer than 3-5 mm from caprone.

IMPORTANT: NO rupture or hole in teflon film in the platinum cathode area are permissible!

Take a new membrane assembly from SPK and, holding the assembly vertically, fill electrolyte with it. Then carefully fit the assembly with electrolyte over the bushing and place the protective bushing.

After membrane assembly/teflon film replacement, perform the steps detailed in para.2.3.3 and 2.3.4.

2.6.5 Platinum electrode cleaning

A need for platinum electrode cleaning in a special solution arises in 6-12 months after the first use. It is not recommended to clean the electrode before this period expires.

To clean the electrode, prepare two solutions.

Solution composition:

- Solution 1: chlorohydric acid (concentrated) – 50 cm³,
distilled water – up to 100 cm³;
- Solution 2: acetic acid (80 to 100 %).

Pour the solutions into vessels making sure that the liquid level is up to 3 mm. Then proceed as follows:

- remove the teflon film;
- wash the sensor with distilled water;
- place the sensor into a vessel with Solution 1 and hold it there for 1 hour;
- wash the sensor with distilled water;
- place the sensor into a vessel with Solution 2 and hold it there also for 1 hour;

IMPORTANT: NEVER PLACE the silver anode in the above solutions!

- wash the sensor with distilled water.

Then proceed to para.2.6.4.2.

Note: After cleaning the platinum electrode and performing the steps as per para.2.6.4.2 and 2.3.3, 2.3.4, the analyzer, when placed in the null solution, can display low negative values over a period from 24 to 48 hours. For the analyzer to resume its normal operation as soon as possible, replace the electrolyte after 24 hours.

2.6.6 Analyzer initial parameter setting

The instrument features a mode for setting the analyzer initial parameters of shift (zero shift) and steepness corresponding to the averaged sensor. This mode enables calibration to be always performed from the fixed initial conditions.

Use this mode when in doubt as to whether the analyzer has operated correctly in performing calibration modes.

2.6.6.1 Zero shift setting

- 1 Switch off the analyzer.
- 2 Press the **CAL** button and, keeping it pressed, switch on the analyzer. After a sound signal release the **CAL** button. The screen will display **c2**.
- 3 Press the **ENTER** button. The **done** capture will briefly appear on the screen and the analyzer will go into the measurement mode. The display will indicate readings in mg/dm^3 with zero shift.

2.6.6.2 Average steepness setting

- 1 Switch off the analyzer.
- 2 Press the **CAL** button and keeping it pressed, switch on the analyzer. After a sound signal release the **CAL** button. The screen will display **c2**. Press the **CAL** button again. The screen will display **c3**.
- 3 Press the **ENTER** button. The **done** capture will briefly appear on the screen and the analyzer will go into the measurement mode. The display will indicate readings in mg/dm^3 corresponding to the average sensor steepness.

Once the analyzer initial parameters are set, proceed to para.2.3.4.

3 MAINTENANCE

Maintenance of the analyzer comprises:

- analyzer calibration by outside air (para.2.3.4) to be carried out every 8 hours;
- analyzer zero calibration (para.2.3.5) to be carried out every three months;
- sensor cycling (para.2.3.3.3) to be carried out when the analyzer has not been in use for more than 24 hours. This operation ensures the highest response rate of the instrument in DOC measurements.

When the conditions described in para.2.5 are fulfilled, the analyzer meets the specifications listed in para.1.3.

4 DELIVERY SET

4.1 The delivery set is shown in Table 4.1.

Table 4.1

Description	Quantity
1 MARK-302T dissolved oxygen analyzer (with sensor, cable length 2 m)	1
2 Flowing vat	1
3 Spare parts kit (to oxygen sensor)	1
4 Tool and accessory kit	1
5 Tool and accessory kit	1
6 Operation Manual	1

APPENDIX A

(reference)

Solubility of 100 % humidity air oxygen in distilled water, depending on the temperature

$$P_{atm} = 101,325 \text{ kPa}$$

Table A.1

t, °C	mg/dm ³									
	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
0	14,62	14,58	14,54	14,50	14,46	14,42	14,38	14,34	14,30	14,26
1	14,22	14,18	14,14	14,10	14,06	14,02	13,98	13,94	13,90	13,87
2	13,83	13,79	13,75	13,72	13,68	13,64	13,60	13,57	13,53	13,49
3	13,46	13,42	13,39	13,35	13,32	13,28	13,24	13,21	13,17	13,14
4	13,11	13,07	13,04	13,00	12,97	12,93	12,90	12,87	12,83	12,80
5	12,77	12,74	12,70	12,67	12,64	12,61	12,57	12,54	12,51	12,48
6	12,45	12,41	12,38	12,35	12,32	12,29	12,26	12,23	12,20	12,17
7	12,14	12,11	12,08	12,05	12,02	11,99	11,96	11,93	11,90	11,87
8	11,84	11,81	11,79	11,76	11,73	11,70	11,67	11,64	11,62	11,59
9	11,56	11,53	11,51	11,48	11,45	11,42	11,40	11,37	11,34	11,32
10	11,29	11,26	11,24	11,21	11,18	11,16	11,13	11,11	11,08	11,06
11	11,03	11,00	10,98	10,95	10,93	10,90	10,88	10,85	10,83	10,81
12	10,78	10,76	10,73	10,71	10,68	10,66	10,64	10,61	10,59	10,56
13	10,54	10,52	10,49	10,47	10,45	10,42	10,40	10,38	10,36	10,33
14	10,31	10,29	10,27	10,24	10,22	10,20	10,18	10,15	10,13	10,11
15	10,08	10,06	10,04	10,02	10,00	9,98	9,96	9,94	9,92	9,90
16	9,87	9,85	9,83	9,81	9,79	9,77	9,75	9,73	9,71	9,69
17	9,66	9,64	9,62	9,60	9,58	9,56	9,54	9,52	9,50	9,49
18	9,47	9,45	9,43	9,41	9,39	9,37	9,36	9,34	9,32	9,30
19	9,28	9,26	9,24	9,22	9,21	9,19	9,17	9,15	9,13	9,11
20	9,09	9,08	9,06	9,04	9,02	8,01	8,99	8,97	8,95	8,93
21	8,91	8,89	8,87	8,86	8,85	8,83	8,81	8,80	8,78	8,76
22	8,74	8,73	8,71	8,69	8,68	8,66	8,64	8,63	8,61	8,60
23	8,58	8,56	8,55	8,53	8,51	8,50	8,48	8,47	8,45	8,43
24	8,42	8,40	8,39	8,37	8,36	8,34	8,32	8,31	8,29	8,28
25	8,26	8,25	8,23	8,22	8,20	8,19	8,17	8,16	8,14	8,13
26	8,11	8,10	8,08	8,07	8,05	8,04	8,02	8,01	7,99	7,98
27	7,97	7,95	7,94	7,92	7,91	7,89	7,88	7,87	7,85	7,84
28	7,83	7,81	7,80	7,78	7,77	7,76	7,74	7,73	7,71	7,70
29	7,69	7,67	7,66	7,65	7,63	7,62	7,61	7,59	7,58	7,57
30	7,56	7,54	7,53	7,52	7,50	7,49	7,48	7,46	7,45	7,44
31	7,44	7,44	7,43	7,42	7,41	7,39	7,38	7,37	7,36	7,35
32	7,33	7,32	7,31	7,30	7,29	7,28	7,26	7,25	7,24	7,23
33	7,22	7,21	7,19	7,18	7,17	7,16	7,15	7,14	7,13	7,11
34	7,10	7,09	7,08	7,07	7,06	7,05	7,04	7,03	7,01	7,00
35	6,99	6,98	6,97	6,96	6,95	6,94	6,93	6,92	6,90	6,89
36	6,82	6,81	6,80	6,78	6,77	6,76	6,75	6,74	6,73	6,72
37	6,71	6,70	6,69	6,68	6,67	6,66	6,65	6,64	6,63	6,62
38	6,61	6,60	6,59	6,58	6,57	6,56	6,55	6,54	6,53	6,52
39	6,51	6,50	6,49	6,48	6,47	6,46	6,45	6,44	6,43	6,42

Table A.1 (Continue)

 mg/dm^3

t, °C	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
40	6,41	6,40	6,39	6,38	6,37	6,36	6,35	6,34	6,33	6,32
41	6,31	6,30	6,29	6,28	6,27	6,26	6,25	6,24	6,23	6,22
42	6,21	6,20	6,19	6,19	6,18	6,17	6,16	6,15	6,14	6,13
43	6,12	6,11	6,10	6,09	6,08	6,07	6,06	6,05	6,04	6,04
44	6,03	6,02	6,01	6,00	5,99	5,98	5,97	5,96	5,95	5,94
45	5,93	5,92	5,92	5,91	5,90	5,89	5,88	5,87	5,86	5,85
46	5,84	5,83	5,82	5,82	5,81	5,80	5,79	5,78	5,77	5,76
47	5,75	5,74	5,74	5,73	5,72	5,71	5,70	5,69	5,68	5,67
48	5,66	5,66	5,65	5,64	5,63	5,62	5,61	5,60	5,59	5,59
49	5,58	5,57	5,56	5,55	5,54	5,53	5,52	5,52	5,51	5,50
50	5,49	5,48	5,47	5,47	5,46	5,45	5,44	5,44	5,43	5,42