ANALYTICAL EQUIPMENT FOR VZOR ECOLOGY AND POWER INDUSTRY

MAPK-501 DISSOLVED HYDROGEN METER

Operation Manual

АЯ 74

Nizhny Novgorod
2010

VZOR enterprise shall be grateful for any proposals and criticisms aimed at improvement of the product.

In the event of any trouble in operating the apparatus you are welcome to report us in writing or by phone.

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C O N T E N T S

1 DESCRIPTION AND OPERATION

1.1 Purpose

1.1.1 Product name and identification
MAPK-501 dissolved hydrogen analyzer
TU 4215-031-39232169-2009 Specifications.

The analyzer is used to measure mass concentration of water-dissolved hydrogen and volume hydrogen concentration in gaseous medium as well as temperature of analyzable medium.

1.1.2 Applications: control of minor dissolved hydrogen concentrations on heat engineering facilities and control of hydrogen content in processes of various industries.

1.1.3 Type of analyzer:

- amperometric;
- with external reference voltage;
- with one sensing element;
- with digital liquid crystal display;
- with automated temperature compensation;
- with flowing dip probe;
- with manual calibration on submersion of the probe into reference hydrogen medium (CGM); and
- automated record of atmospheric pressure on calibration.

1.2 Main Parameters

1.2.1 By resistance to climatic load the analyzer version meets GOST R 52931-2008 − B4.

1.2.2 By resistance to mechanical action the analyzer version meets GOST R 52931-2008 − L1.

1.2.3 Convertor unit protection level ensured by enclosure meets GOST 14254-96, − IP30.

1.2.4 By resistance to atmospheric pressure the analyzer version meets GOST R 52931-2008 – P1 (atmospheric pressure from 84 to 106.7 kPa).

1.2.5 Parameters of analyzable medium

1.2.5.1 Analyzable medium temperature, °C ……………………... + 5 to + 50.

1.2.5.2 Analyzable medium pressure, MPa, max. ……………….....……….. 0.

1.2.5.3 Analyzable aqueous medium pH …………………………….... 4 to 12.

1.2.6 Operating conditions:

1.2.6.1 Ambient air temperature, °C ………………………………. + 1 to + 50.

1.2.6.2 Ambient air relative humidity at temperature of + 35 °C and below without moisture condensation, %, max …………………………………………… 80.

1.2.6.3 Atmospheric pressure, kPa (mm of Hg) ……………….. 84,0 to 106,7 (from 630 to 800).

1.2.7 The analyzer is calibrated against reference hydrogen medium.

1.2.8 The analyzer is powered up from a self-contained power supply with voltage from 2,4 to 3,4 V (two AA alkaline dry cells).

1.2.9 Analyzer power consumption (at rated supply voltage of 3,0 V), mW, max ……………………………………………………………………………… 10.

1.2.10 The analyzer remains in-spec characteristics after dry cell replacement and calibration.

1.2.11 Dimensions and weight of analyzer components are shown in Table 1.1.

Table 1.1

Name and identification of components	Overall dimensions, mm, max	Maximum weight, kg
Convertor unit BP53.01.000	84 × 160 × 38	0,30
hydrogen probe (without a cable) BP53.02.000	∅30 × 135	0,10

1.2.12 Conditions for transportation in shipping containers meet GOST R 52931-2008:

– sinusoidal vibration with 5-35 Hz frequency, 0.35 mm shift amplitude in the direction shown by the "UP" sign on a container;

– temperature, °C …………………...– 20 to + 55;

– air relative humidity at 35 °C, % …..........……………………………… 95 ± 3.

1.3 Specification

1.3.1 Analyzer measurement range:

– for measuring hydrogen mass concentration, μg/dm^3 ………………….. 0 to 2000;

– for measuring hydrogen percentage, % by volume ……………….. 0 to 100.

1.3.2 The range of analyzer allowable basic absolute accuracy at analyzable medium temperature of (20,0 ± 0,2) °C and ambient air temperature of (20 ± 5) °C:

– for measuring mass concentration, μg/dm^3 .... ± (1,0 + 0,035*C*);

– for measuring volume concentration, % ....… ± (0,06 + 0,035*A*),

where *C* is measurable hydrogen mass concentration in analyzable aqueous medium, μg/dm^3;

A is measurable hydrogen percentage in analyzable gaseous medium at humidity of 100 %, % by volume.

1.3.3 The range of analyzer allowable complementary absolute accuracy governed by changes in analyzable medium temperature per each ± 5 °C of normal (20,0 ± 0,2) °C within the operating temperature range from + 5 to + 50 °C:
- for measuring mass concentration, μg/dm^3 .... ± (0,3 + 0,013*C*);
- for measuring volume concentration, % .... ± (0,02 + 0,013*A*).

1.3.4 The range of analyzer allowable complementary absolute accuracy governed by changes in ambient air temperature per each ± 10 °C of normal (20 ± 5) °C within the operating temperature range from + 5 to + 50 °C:
- for measuring mass concentration, μg/dm^3 .... ± 0,0075*C*;
- for measuring volume concentration, % .... ± 0,0075*A*.

1.3.5 Measuring range of analyzable medium temperature, °C....... 0 to + 50.

1.3.6 The range of analyzer allowable basic absolute accuracy on measuring analyzable medium temperature at ambient air temperature of (20 ± 5) °C, °C.. ± 0,3.

1.3.7 The range of analyzer allowable complementary absolute accuracy on measuring analyzable medium temperature governed by changes in ambient air temperature per each ± 10 °C of normal (20 ± 5) °C within the operating range from + 1 to + 50 °C, °C .... ± 0,2.

1.3.8 Allowable setting time on measuring hydrogen mass concentration or percentage $t_{0.9}$, min .... 5.

1.3.9 Allowable analyzer setting time for measuring hydrogen mass concentration or percentage t_y, min .... 40.

1.3.10 Allowable setting time for measuring analyzable aqueous medium temperature $t_{0.9}$, min .... 7.

1.3.11 Allowable setting time for measuring analyzable aqueous medium temperature t_y, min .... 20.

1.3.12 Instability of analyzer readings for a period of 8 h, maximum:
- for measuring mass concentration, μg/dm^3 .... ± 0,0175*C*;
- for measuring volume concentration, % .... ± 0,0175*A*.

1.4 Analyzer components

The analyzer is composed of:
- a convertor unit; and
- dissolved hydrogen probe with a cable 2 m long.

1.5 Design and operation

1.5.1 Analyzer general data

MAPK-501 dissolved hydrogen analyzer is a small-size microprocessor instrument used to measure mass concentration of hydrogen dissolved in water and hydrogen volume concentration in gaseous medium, and also temperature of analyzable medium.

Hydrogen mass concentration measured in $\mu g/dm^3$, hydrogen volume concentration measured in % or temperature measured in Celsius degrees (depending on measurement conditions) are shown on a digital liquid crystal display (hereafter display). The least significant digit for measuring dissolved hydrogen concentration is 0,1 $\mu g/dm^3$, and for measuring hydrogen volume percentage it is 0,01 %. The least significant digit for measuring temperature is 0,1 °C.

Analyzer calibration is undertaken against:

- hydrogen-free ("zero") medium (semi-automatic); and
- reference hydrogen medium of 100 % humidity (manual).

A built-in atmospheric pressure sensor is used to measure atmospheric pressure in analyzer hydrogen medium calibration.

1.5.2 Analyzer operating principle

To measure hydrogen content, the analyzer is furnished with amperometric sensor working as a closed-type polarographic cell.

Electrodes are submerged in electrolyte solution which is isolated from the measurable medium by a membrane permeable for hydrogen and impermeable for liquids and water vapors. Hydrogen from analyzable medium penetrates through a membrane and diffuses through a thin electrolyte layer between the anode and the membrane and undergoes an electrochemical reaction on the anode surface which is polarized by external voltage between electrodes. In this case the sensor generates a DC signal which at constant temperature is proportional to concentration of hydrogen dissolved in the controllable medium.

Hydrogen probe sensitivity (proportionality factor) rises as the controllable medium temperature increases. To compensate for this dependence, the analyzer is furnished with automatic temperature compensation function that involves a temperature sensor which shares the same enclosure with the hydrogen probe.

1.5.3 Analyzer design

The analyzer is shown in Fig. 1.1.

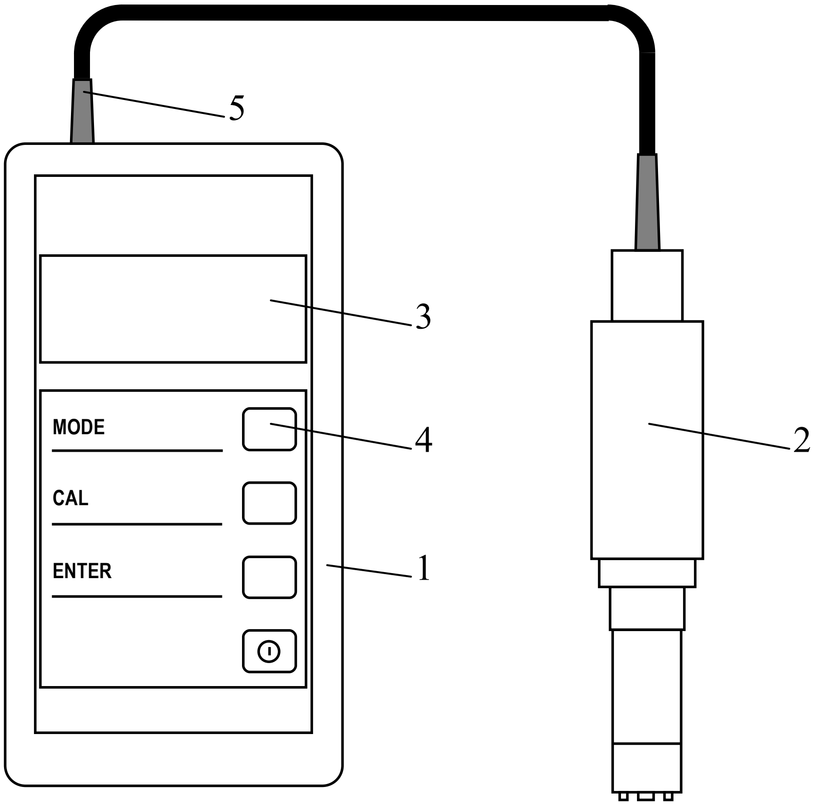


Fig. 1.1

The convertor unit 1 (CU) enclosed in a plastic tight casing transforms signals from the hydrogen probe 2 into display of measurement results.

The CU front panel carries:

– a display screen 3 to show measured mass concentration of hydrogen dissolved in water; mass hydrogen concentration in gaseous medium or temperature (depending on selected mode of measurement), dry cell discharge indication, and current time indication (in the OFF position); and

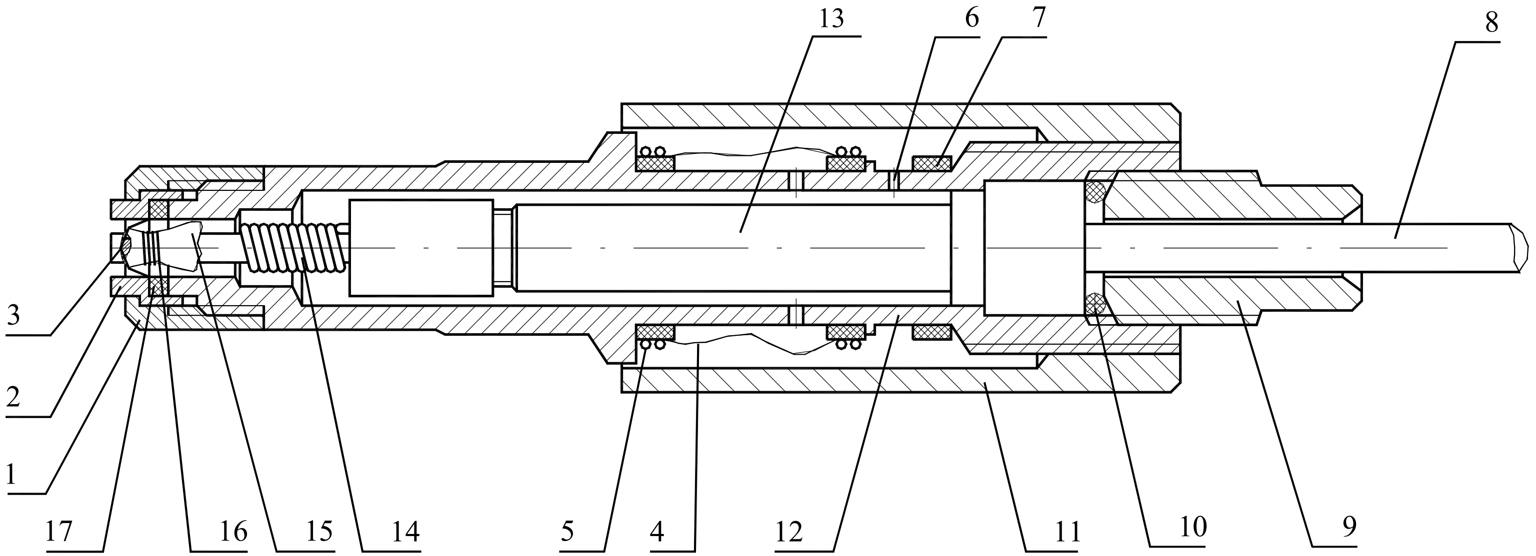
– buttons 4.

The convertor unit back panel bears a cover of the dry cell compartment.

The upper end face of the analyzer convertor unit carries a tight hydrogen probe 2 cable entry 5.

Fig. 1.2 shows main components of the hydrogen probe enclosed in a polypropylene casing.

The platinum anode 3 is embedded into the end face of electrode-holding glass tube, while a silver cathode 14 is wound round the tube. The holding tube and cable 8 are tightly embedded into the inner casing 13 nested in the main casing 12 and secured by a nut 9 with a seal ring 10.



1 – coupling nut, 2 – castle bushing, 3 – platinum anode, 4 – diaphragm, 5 – diaphragm clamp, 6 – hole for filling electrolyte, 7 – rubber ring, 8 – cable, 9 – nut, 10 – rubber ring, 11 – protective cup, 12 – main casing, 13 – inner casing, 14 – silver cathode, 15 – Teflon film, 16 – nylon threads, 17 – rubber ring and membrane assembly.

Fig. 1.2 – Hydrogen probe

The holding tube bears a Teflon film 15 of 15 μm thickness secured with nylon threads 16. The film ensures a constant gap between the anode and the membrane. The membrane and a rubber ring 17 are inserted into the castle bushing 2. The castle bushing is located on the main casing and secured with a coupling nut 1.

The main casing bears a diaphragm 4 used to equalize pressure outside and inside the probe. Clamps 5 retain the diaphragm on the main casing and make the probe inner space with electrolyte tight.

Electrolyte is filled through holes 6 in the main casing which are closed with rubber rings 7 in the working position.

A protection cup 11 is used to prevent diaphragm damage and incidental ring 7 displacement, and also serves as a decorative element.

1.5.4 Functions of the buttons on the convertor unit front panel

As shown in Fig. 1.1, the following is mounted on the front panel:

– "  " is used to switch on and off the analyzer. When the analyzer is ON, the display shows the measured concentration of dissolved hydrogen, hydrogen percentage or temperature;

– "**MODE**" button is used to change analyzer modes. When the button is depressed consecutively, the display shows measured concentration of dissolved hydrogen, temperature or hydrogen percentage and "***μg/dm^3***", "**°C**" or "**%**" indications on the right side of the display;

– "**CAL**" button is used to select an analyzer calibration mode. When the button is depressed consecutively, the display alternately shows "***cdH0***" indication meaning analyzer zeroing and "***cdH I%***" indication meaning calibration against the reference hydrogen medium;

– "**ENTER**" is used to confirm selection of the calibration mode and to terminate calibration.

1.6 Instruments, tools and appliances

1.6.1 Analyzer maintenance additionally requires:

– hydrogen-nitrogen control gas mixture (CGM) with hydrogen concentration of 40 to 100 % vol.; and

– thermometer with 0,1 °C division value.

2 INTENDED USE

2.1 Operating limitations

2.1.1 The analyzer is mostly used to measure hydrogen mass concentration in deaerated water and hydrogen percentage in gaseous medium.

2.1.2 The analyzer must be mounted so as to prevent water ingress onto the convertor unit.

2.1.3 When using the analyzer, protect the hydrogen probe against any shock, since its design uses glass.

2.2 Safety precautions

2.2.1 Before using the dissolved hydrogen analyzer, the personnel must carefully read this manual and safety rules for handling high-pressure bottles.

2.2.2 The analyzer meets safety requirements of Class III according to GOST R 52319-2005. Rated supply voltage varies from 2,2 to 3,4 V. Grounding is not required.

2.2.3 Analyzer electromagnetic compatibility meets requirements of GOST R 51522-99 for class B equipment.

2.3 Pre-starting procedure

Unpack the analyzer on receipt, check the components and make sure that the products are free of damage.

If the analyzer was kept in cold environment, hold it at room temperature for at least 1 hour and then start pre-starting operating procedures.

2.3.1 Connection of power supply

To connect power supply, remove the dry cell compartment cover on the back panel of the convertor unit. Install two AA alkaline dry cells according to the marking in the battery compartment. Close the dry cell compartment cover.

Switch on the analyzer. The display will show dissolved hydrogen concentration in μg/dm^3 or hydrogen percentage in %, or temperature in °C.

NOTICE: STRICTLY OBSERVE POLARITY when connecting power supply. Otherwise this may cause analyzer failure!

If the display shows " [+ −] ", replace AA alkaline dry cells.

2.3.2 Actuation of time indicator

With power supply in the dry cell compartment, the analyzer in the OFF position can display time.

Time display may be switched on or off, if two "**MODE**" and "**ENTER**" buttons are concurrently depressed with the analyzer in the OFF position. The display shows time "**HH.MM**" and the point between hour and minute indications blink at 1 s interval.

To correct time proceed as follows:

– press the "**CAL**" button, and minute indication on the display starts blinking;

– set minutes using the "**MODE**" and "**ENTER**" buttons;

– press the "**CAL**" button, and hour indication on the display starts blinking;

– set hours using the "**MODE**" and "**ENTER**" buttons;

– press the "**CAL**" button to terminate time correction, and the analyzer will enter the time indication mode.

2.3.3 DH probe preparation

The DH probe is supplied as part of the analyzer complete set in "dry" condition and is to be filled with electrolyte included in the scope of supply as prescribed in 2.6.3 and submerged in distilled water for at least 24 h.

Two AA alkaline dry cells must be installed in the convertor unit. Irrespective of whether the analyzer is ON or OFF, polarization voltage shall be applied to the probe to shape the electrode system.

After replacement of the membrane or teflon film, the probe, prior to calibration, should be held in distilled water for at least 24 h to stabilize tension of the membrane and teflon film.

2.3.4 Probe check against the medium with zero hydrogen content (air)

Air check that makes it possible to determine the probe response time and its ability to go to "zero" is the main operational check.

Air check must be carried:

- after replacement of the membrane or teflon film;
- if the analyzer readings seem to be doubtful; and
- after long analyzer downtime.

For air check, remove the probe from water, shake water drops off the membrane and place it in air at 15-30° angle to the horizontal as shown in Fig. 2.1.

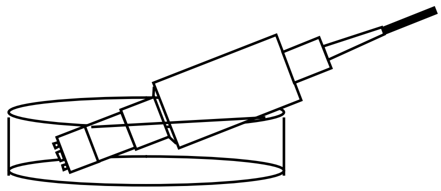


Fig. 2.1

Record analyzer reading in a 40-min period.

Readings in air within ±1 μg/dm^3 are normal. In this case analyzer desired metrological characteristics are ensured. Then proceed with calibration: against CGM GSO with known hydrogen content in % vol. or against the solution with known mass concentration of dissolved hydrogen (2.3.6).

If the readings lie within ± 3 μg/dm^3 after 40 min, undertake air calibration (2.3.5) which makes it possible to set analyzer zero reading.

If display readings are beyond ± 3 μg/dm^3, refer to 2.5 of the Operation Manual (Troubleshooting).

2.3.5 Analyzer "zero" calibration

Analyzer "zero" calibration allows for compensation within small ranges (from minus 3,0 to plus 3,0 μg/dm^3) of readings corresponding to probe residual "zero" current.

For air calibration remove the probe from water, shake water drops off the membrane and place it in air at 15-30° angle to the horizontal as shown in Fig. 2.6.

Switch on the analyzer.

Keep the probe in air for at least 40 min.

To set analyzer "zero", proceed as follows.

1 Press the "**CAL**" button. The analyzer display will show "***cdH0***".

2 Press the "**ENTER**" button. The analyzer display will show Z_0 in the "zero" solution without "zero" correction, for instance, "***c002.0 µg/dm³***".

3 In at least 8-second period, press the "**ENTER**" button again. The display will show "***donE***" for a short time, and the analyzer will enter the measurement mode. The analyzer display will show the following readings in air after setting the analyzer "zero":

- 000,0; if $-003,0\ \mu g/dm^3 \le Z_0 \le 003,0\ \mu g/dm^3$;
- $Z_0 - 003,0$; if $Z_0 > 003,0\ \mu g/dm^3$;
- $Z_0 + 003,0$; if $Z_0 < -003,0\ \mu g/dm^3$.

Note – Analyzer "zero" setting may be put off till step 3 by pressing the "**MODE**" button. The analyzer will enter the measurement mode retaining calibration factor values from the previous calibration.

2.3.6 Analyzer hydrogen calibration

Hydrogen calibration is required:

- on probe receipt (after filling electrolyte and stabilization of the electrode system, 2.3.3);
- after replacement of the membrane or teflon film;
- if analyzer readings seem to be doubtful; and
- on a quarterly basis.

To increase measurement accuracy, we recommend that analyzer calibration be undertaken against CGM GSO, or a solution with known mass concentration of dissolve hydrogen and hydrogen content close to values to be measured.

To reduce analyzer complementary error due to changes in analyzable medium temperature, probe temperature in hydrogen calibration must be close to analyzable medium temperature.

Preferably, hydrogen calibration should be undertaken at room temperature against CGM GSO with hydrogen concentration from 40 to 100 % vol.

Prompt hydrogen calibration may involve the calibrator from the analyzer spare parts kit.

2.3.6.1 Analyzer calibration against CGM GSO

For calibration against CGM GSO assemble the package as shown in Fig. 2.2.

Fill the vessel with distilled water of room temperature.

In the vessel install:

– a probe with a tip (for instance, CT-18 PVC tube; $\varnothing_{internal}16\times2$, L = 60 mm) extended 30-35 mm from the probe end. The probe must be placed in the vessel at 60-70° angle to the horizontal;

– a bent capillary tube connected to the CGM bottle outlet;

– hold the probe with the tip in the vessel for at least 30 min;

– use a capillary tube to feed CGM from the bottle to the probe membrane. Using a rotameter, set the CGM feed rate so that an air bubble in the cup is changed each 3-5 s. Wait till readings are stable.

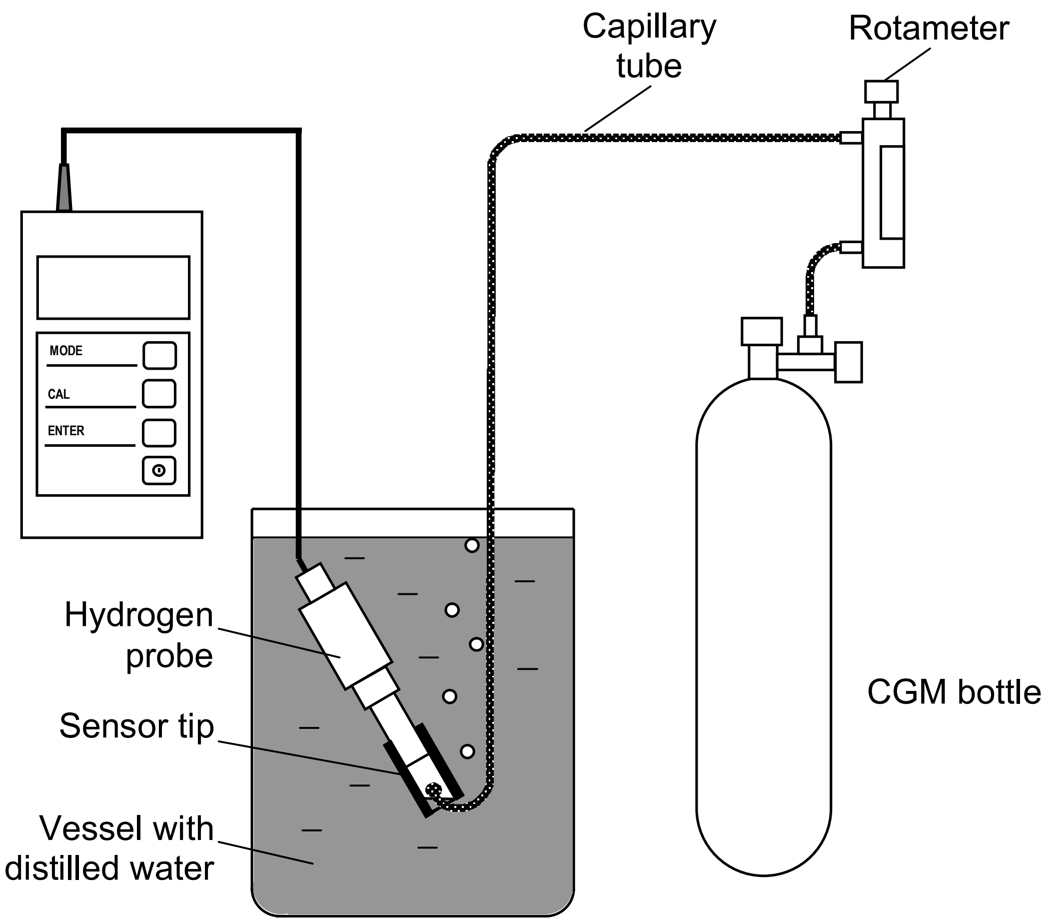


Fig. 2.2 – Analyzer calibration against CGM GSO

For hydrogen calibration using CGM proceed as follows.

1 Press the "**CAL**" button twice. The display will show "***cdH I%***" meaning that the hydrogen calibration mode is selected.

2 Press the "**ENTER**" button. The display will show the value in % vol. measured by the analyzer, for instance "***c93.5 %***".

3 Wait till readings are stable for at least 8 seconds and using the "**MODE**" (increase) and "**CAL**" (decrease) buttons, set concentration of CGM in the bottle in % vol. on the display ("***cXX.X %***").

4 Press the "**ENTER**" button once again. The display will show "***donE***" for a short time, and the analyzer will enter the measurement mode. It means that the hydrogen calibration mode is over and the analyzer is calibrated.

On completion of hydrogen calibration, the analyzer is available for use.

Note – Analyzer hydrogen calibration may be put off till step 3 by pressing "**ENTER**" button. The display shall show "***cdH I%***". If the "**MODE**" button is depressed, the analyzer enters the measurement mode retaining calibration factor values from the previous calibration.

2.3.6.2 Prompt analyzer calibration may involve the calibrator from the analyzer spare parts kit

For hydrogen calibration using the calibrator the following is required:

– switch on the analyzer;

– loosen nut;

– push the analyzer probe measuring element into the calibrator socket against stop as shown in Fig. 2.3;

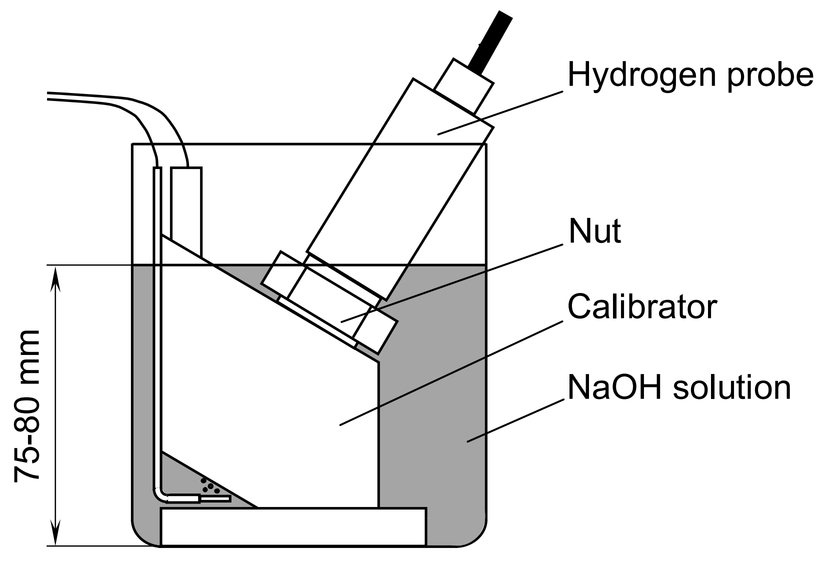


Fig. 2.3 – Analyzer calibration in the calibrator

– tighten the nut;

– place the calibrator into a 0,5-1 dm^3 container;

– fill the container with NaOH solution of 4 g/dm^3 concentration to 75-80 mm mark;

– connect the calibrator to the power supply socket. In this case gas shall release on the calibrator electrodes;

– 1 hour after setting the probe measuring element into the calibrator socket visually inspect and make sure that hydrogen bubble is available in the bottom part of the calibrator socket, and disconnect the calibrator from the power supply.

Wait for stable readings and make hydrogen calibration.

1 Press the "**CAL**" button twice. The display will show "***cdH I%***" meaning that hydrogen calibration mode is selected.

2 Press the "**ENTER**" button. The display shows value in % vol. measured by the analyzer, for instance "***c93.5 %***".

3 In at least 8 s using the "**MODE**" (increase) and "**CAL**" (decrease) buttons set 90 % vol. on the display ("***c90.0 %***").

4 Press the "**ENTER**" button once again. The display shall show "***donE***" for a short time and the analyzer enters measurement mode. It means that the hydrogen calibration mode is over and the analyzer is calibrated.

Note – Analyzer hydrogen calibration may be postponed till step 3 by pressing the "**ENTER**" button. The display shall show "***cdH I%***". If the "**MODE**" button is depressed, the analyzer enters the measurement mode retaining the calibration factor values from the previous calibration.

2.3.6.3 Analyzer calibration against solution with known mass concentration of dissolved hydrogen

Calibration against solution with known mass concentration of dissolved hydrogen requires, for instance, a reference dissolved hydrogen analyzer. In this case mass concentration of dissolved hydrogen is to be measured by the reference and working analyzers using the same solution.

Wait for stable readings of both analyzers and start calibration.

To undertake hydrogen calibration with a reference analyzer, proceed as follows.

1 Switch off the analyzer.

2 Press the "**CAL**" button and keeping it depressed switch on the analyzer.

3 Press the "**CAL**" button to select "***cdH I µg/dm³***" mode.

4 Press the "**ENTER**" button. The display will show the value in $µg/dm^3$ measured by the analyzer, for instance "***c023.3 µg/dm³***".

5 In at least 8 sec using the "**MODE**" and "**CAL**" buttons, set the value on the display read by the reference analyzer, for instance "***c020.0 µg/dm³***".

6 Press the "**ENTER**" button once again. The display shall show "***donE***" for a short time, and the analyzer will enter the measurement mode. It means that hydrogen calibration mode is over and the analyzer is calibrated.

Note – Analyzer hydrogen calibration may be put off till step 5 by pressing the "**ENTER**" button. The display shall show "***cdH I µg/dm³***". If the "**MODE**" button is depressed, the analyzer will enter the measurement mode retaining calibration factor values from the previous calibration.

2.4 Pre-measurement operations

A flowing vat is used at a flow rate from 0,07 to 0,60 dm^3/min.

To set up the hydrogen probe in the cell proceed as follows:

- loosen nut;
- push the probe into the flowing vat as shown in Fig. 2.4 as deep as possible (against stop);
- tighten the nut.

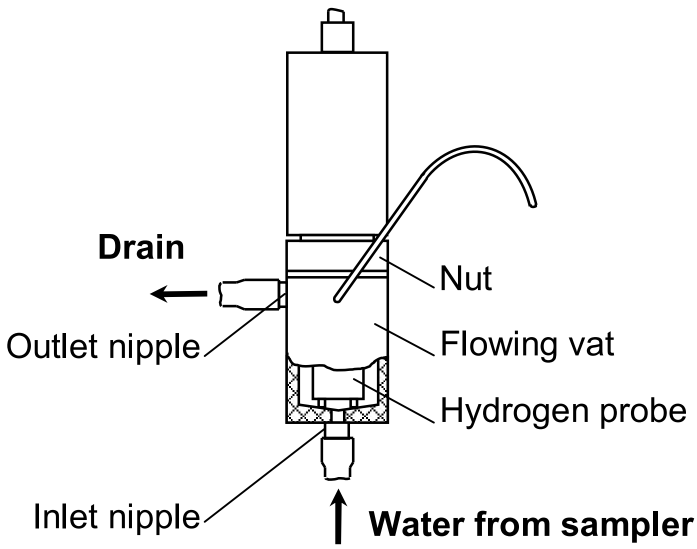


Fig. 2.4 – Probe disposition in the flowing vat in measurements

Before removing the probe from the flowing vat, remove the hose from the outlet nipple and loosen the nut.

The flowing vat may be used for storage and transportation of the probe. To do so, leave water in the cell and connect the cell hoses.

2.5 Measurement procedure

2.5.1 Measurement of dissolved hydrogen mass concentration using a flowing vat

Place a flowing vat in a fairly vertical position.

Connect the inlet nipple of flowing vat with a probe inside to the line with analyzable water using a flexible hose.

Check all connections. The sampling line is to be leak-proof to prevent ingress of outside air.

Feed analyzable water. Make sure that the water flow and the probe membrane are both free of air bubbles.

Air bubbles in the hose bends, on the probe membrane or in water supply line may produce significantly inaccurate results. One of the symptoms of air bubbles consists in analyzer readings failure to reach stability and their tendency to slowly and continuously drop. Such drop of analyzer readings may last 1-2 h.

To remove bubbles from the membrane, shake gently the cell with the probe.

To remove bubbles from the sampler line, we recommend that flow be promptly enhanced for 10-20 s and then returned to normal rate.

Remove the probe from the cell for the period of rapid flow.

If measurements are undertaken continuously, take measures to prevent probe overheat (over 70 °C).

Flow rate through the cell is to be maintained within the range from 0,07 to 0,60 dm^3/min. High water flow rate may cause analyzer unstable readings. Too high flow rate may lead to probe membrane damage.

2.5.2 Measurement of dissolved hydrogen mass concentration in laboratory conditions

To carry out measurements in laboratory conditions, fill analyzable water to an appropriate container and ensure that the analyzable water has flow rate in relation to the probe membrane at least 5 cm/s. To do so, we recommend to use a magnetic stirrer.

2.5.3 Measurement of hydrogen percentage

To measure hydrogen percentage, a facility similar to that in Fig. 2.2 may be used except that an analyzable gas shall be applied to the hydrogen probe membrane instead of CGM GSO. Using a rotameter set the gas flow rate so that an air bubble in the cup is changed each 3-5 s. Wait till readings are stable, and record them as measurement results.

<u>Note</u> – To save metrological characteristics of the hydrogen probe, we recommend that the probe be left in gaseous medium for no more than 8-10 h. In the event of long outages the hydrogen probe must be stored with the membrane submerged in distilled water.

2.5.4 Measurement of water temperature

For measuring temperature, the hydrogen probe must be fully submerged in water.

For temperature measurements press the "**MODE**" button to select the "**t °C**" temperature measurement mode.

Wait till analyzer readings reach stability, and record them as measurement results.

2.6 Troubleshooting

2.6.1 Typical analyzer troubles and remedies are set out in Table 2.2.

Table 2.1

Trouble	Probable cause	Remedy
1 With power ON, display shows no indications or readings	Poor contact in the dry cell compartment	Open the dry cell compartment and clean contacts
	Supply voltage is below the allowable value	Para 2.3.1. Replace the dry cells

Table 2.1 (continued)

Trouble	Probable cause	Remedy
2 With power ON, display shows all or random segments and signs	Discharged dry cells	Para 2.3.1. Replace the dry cells
3 On checking "zero" point in the range of measurements analyzer readings go beyond ± 3,0 μg/dm^3	Torn or pierced membrane, affected probe tightness	Para 2.6.6, 2.6.4. Replace the membrane and electrolyte
	Moisture ingress into the convertor unit	Dry up the convertor unit for 3-4 days
	Stretched membrane	Para 2.6.6. Replace the membrane assembly
	Broken (cracked) probe electrode glass tube-holder	To be repaired at factory
4 On analyzer hydrogen calibrating, the display indicates "***EGL***" meaning that the probe current is below the rated value	No electrolyte	Para 2.6.3. Fill electrolyte
	Dirty membrane	Para 2.6.2. Clean the membrane
	Dry membrane	Make membrane wet for 2-3 days without disassembly
	Defective membrane	Para 2.6.6. Replace membrane assembly
	Analyzer probe is in a medium other than hydrogen	Place the probe in hydrogen medium
5 Electrolyte leaks rapidly	Torn membrane	Para 2.6.6. Replace membrane assembly
6.1 Analyzer readings change rapidly and are unstable. 6.2 On analyzer hydrogen calibrating the display indicates "***EGh***" meaning that probe current is in excess of the rated value.	Torn membrane	Para 2.6.6. Replace membrane assembly
	Dirty electrolyte	Para 2.6.4. Replace electrolyte
	Moisture ingress into the measuring block	Dry up the convertor unit for 3-4 days
	Torn teflon film	Para 2.6.7. Replace the Teflon film
	Analyzer probe is in a medium other than hydrogen	Place the probe in hydrogen medium
7 Slow response to change in hydrogen concentration	Dirty membrane	Para 2.6.2. Clean the membrane

Table 2.1 (continued)

Trouble	Probable cause	Remedy
8 During measurements the display shows a dash-line " **- - -** " meaning that display reading digit capacity is faulty: readings are in excess of 1999 $\mu g/dm^3$ or below minus 1999 $\mu g/dm^3$	Torn membrane	Para 2.6.6. Replace the membrane assembly
	Dirty electrolyte	Para 2.6.4. Replace electrolyte
	Moisture ingress into the measuring block	Dry up the convertor unit for 3-4 days
	Torn teflon film	Para 2.6.7. Replace the teflon film
	Operator's errors on analyzer calibration	Para 2.6.8. Set initial analyzer parameters
	Analyzer failure	To be repaired at factory
9 During measurements display shows "***Et***" message	Faulty temperature measuring channel (break in heat probe)	To be repaired at factory
10 Display shows "***EFG***" message	Erroneous record in EEPROM memory	To be repaired at factory
11 Analyzer readings change rapidly and are unstable during measurements in the flowing vat	Very high flow rate in the flowing vat	Set water flow rate in the flowing vat from 0,07 to 0,60 dm^3/min

2.6.2 Membrane cleaning

To clean the probe membrane, use a piece of cotton wool wet with alcohol. The probe membrane may be submerged in a weak solution (2 %) of sulfuric acid for about 1 h and then flushed under running water.

2.6.3 Electrolyte filling (refilling)

The hydrogen probe from the analyzer complete set is delivered dry and before use it is to be filled with electrolyte from the delivery set.

To do so, as shown in Fig 2.5, proceed as follows:

– draw electrolyte into a syringe;

– turn the coupling nut counter clockwise, remove it and moisten the membrane inside and the rubber ring with electrolyte;

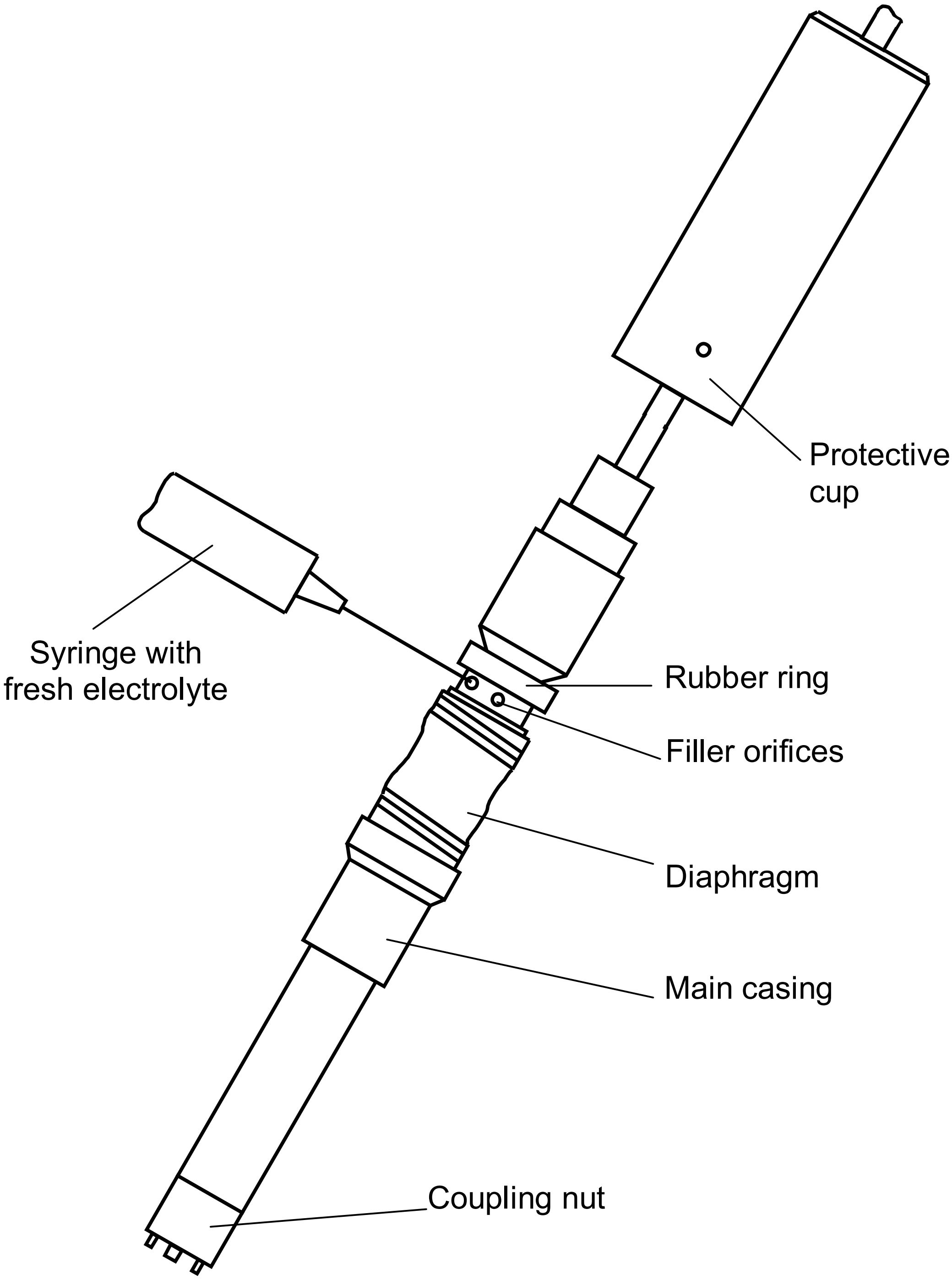


Fig. 2.5 − Electrolyte filling (refilling)

- turn the coupling nut clockwise against stop to push membrane to the platinum cathode;
- loosen the protective cup;
- shift the protective cup from the probe casing to the connecting cable;
- inject 4 cm^3 of electrolyte through one of the filler orifices in the probe casing using a syringe. To allow electrolyte to reach electrodes, shake the probe a few times;
- displace the rubber ring so that it covers both filler orifices; and
- tighten the protective cup.

Then switch on the analyzer and submerge the probe membrane into distilled water for 24 h.

2.6.4 Electrolyte replacement

Electrolyte is to be replaced when it is dirty, or tightness of the membrane or the diaphragm is affected. The symptoms comprise unstable analyzer readings and high readings with the probe in air.

To do so, (after replacement of the membrane or diaphragm, when required) proceed as follows:

– loosen the protective cup as shown in Fig. 2.6;

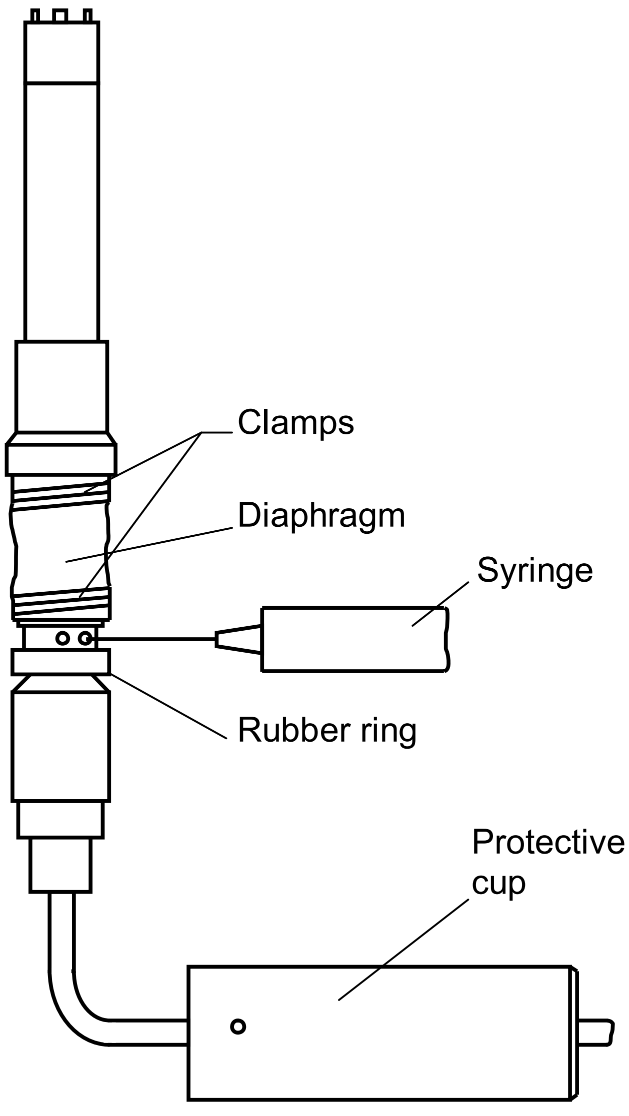


Fig. 2.6 – Replacement of electrolyte

– displace the rubber ring to uncover two electrolyte filler orifices;

– turn the probe so that the membrane assembly is in the up position, and draw off old electrolyte thru one of the filler orifices using a syringe; and

– fill fresh electrolyte as described in 2.6.3.

2.6.5 Diaphragm replacement

2.6.5.1 Damage to diaphragm tightness may cause electrolyte outflow or contamination.

To replace the diaphragm proceed as follows:

– loosen the protective cup as shown in Fig. 2.6;

– inspect the diaphragm visually and remove it if visible mechanical damages are detected (cracks, orifices);

– install a new diaphragm from the spare parts kit and smooth it out so that the diaphragm tightly fits on silicone sealing rings;

– fold the end of the thread and place it along the diaphragm as shown in Fig. 2.7a;

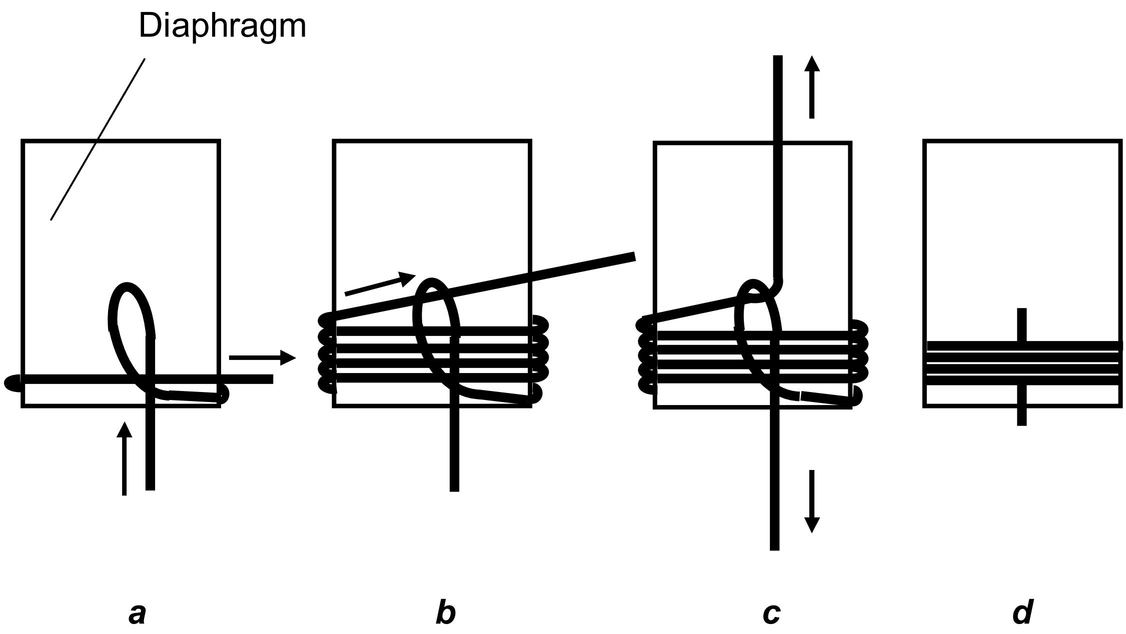


Fig. 2.7

– wind 5-6 coils round the loop near to the silicone ring and then insert the thread end through diaphragm loop as shown in Fig. 2.7*b*;

– pull the thread end to shift the resultant knot under diaphragm thread coils as shown in Fig. 2.7*c*;

– cut off excessive length of the diaphragm thread as shown in Fig. 2.7*d*;

– fix the diaphragm in a similar way near the second silicone ring; and

– tighten the protective cup.

2.6.6 Replacement of membrane

The membrane should be replaced in the event of damage (cracks, stretching) of which symptoms are as follows:

– unstable analyzer readings;
– high readings in air; and
– slow response in measurements of hydrogen concentration.

To replace the membrane, proceed as follows:

– turn the probe so that the membrane assembly is in the up position in order to prevent electrolyte outflow during disassembly thereof;
– loosen the coupling nut remove the old membrane assembly (castle bushing with a rubber ring and membrane);
– make sure that teflon film is free of mechanical defects (holes, cracks, crumples) and tightly fits on the platinum cathode;
– replace defective teflon film in compliance with 2.6.7;
– install a new membrane assembly from the spare parts kit in the coupling nut;
– moisten the membrane and rubber ring inside with electrolyte;
– tighten the nut onto the probe casing against stop; and
– fill electrolyte in compliance with 2.6.3.

Then hold the probe in distilled water with the analyzer on for at least one hour and carry out operations outlined in 2.3.4.

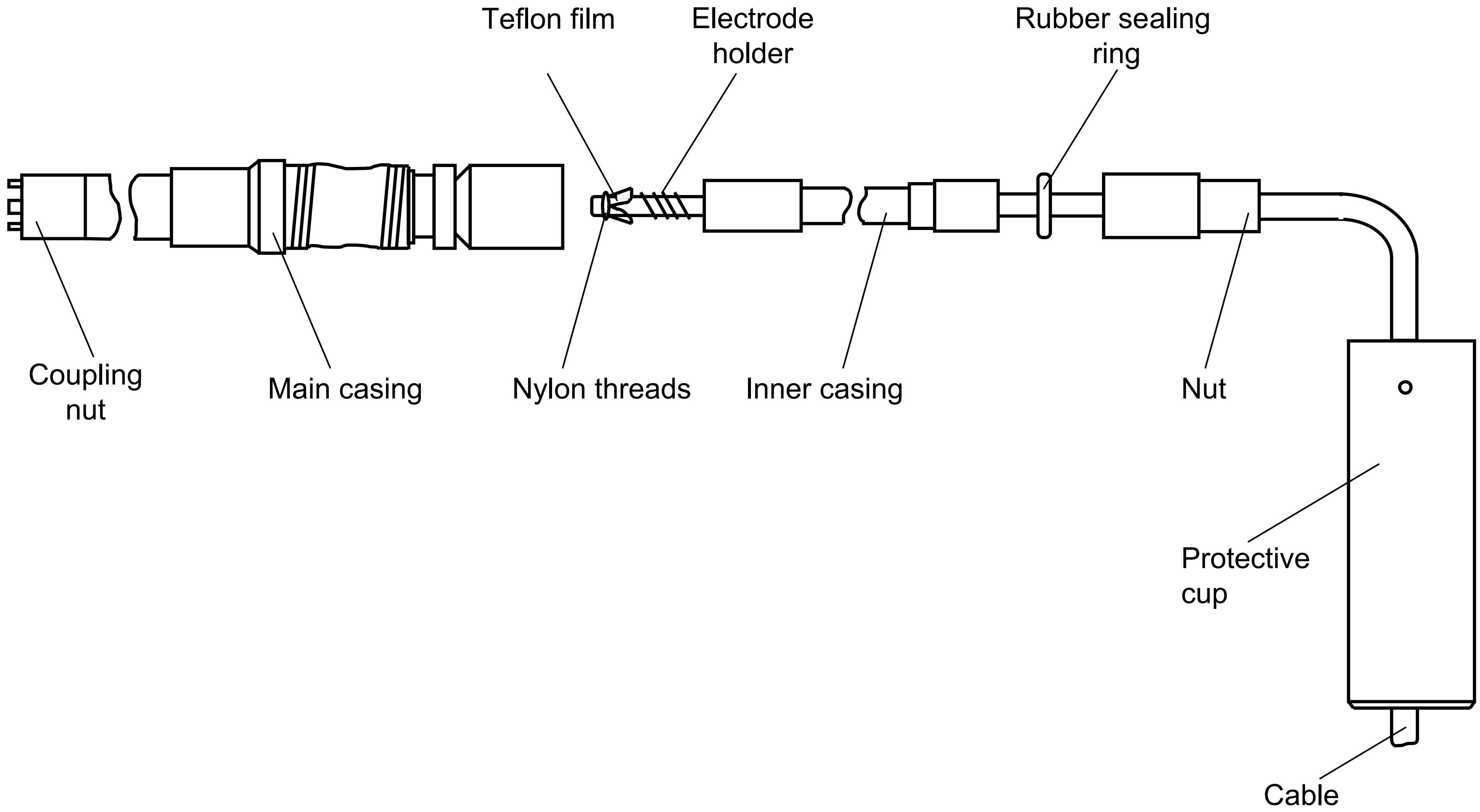
2.6.7 Replacement of teflon film

The teflon film is replaced:

– if it has visible defects; and
– if replacement of the membrane assembly or diaphragm failed to ensure normal operation of the probe.

To replace the teflon film, proceed as follows:

– loosen the protective cup as shown in Fig. 2.8;
– screw out the nut with flattened surfaces;
– carefully remove the rubber sealing ring using a pincers;
– carefully remove the inner casing out of the main casing and drain electrolyte;
– remove old teflon film;


Teflon film
Electrode holder
Rubber sealing ring
Coupling nut
Main casing
Nylon threads
Inner casing
Nut
Protective cup
Cable

[redacted]

– inspect probe electrodes. The platinum anode embedded into a glass tube is to be black. The silver cathode wound round the tube is to be of grey color.

WARNING: DO NOT touch the platinum anode! Wash with distilled water, if required!

– take teflon film from the spare parts kit and apply it onto the anode surface. Do not move the film on the electrode since special black coating applied onto the anode may be easily damaged; and

– press film edges to the glass tube surface and holding them by hand wind up and 5-6 coils of nylon thread and make 2-3 knots. The film shall be tightly pressed to the cathode.

WARNING: TEFLON FILM MUST BE FREE FROM TEARS AND HOLES!

The probe shall be assembled in the following way:
– insert inner casing into the main casing;
– set up the rubber sealing ring;
– tighten the nut;
– fill electrolyte according to 2.3.3; and
– tighten the protective cup.

After the overhaul, hold the probe in distilled water with the analyzer on for at least 3 h and then carry out operations stated in 2.3.4.

2.6.8 Setting of initial analyzer parameters

A provision is made in the instrument for setting the analyzer initial parameters:
– shifting (zero hydrogen shifting); and
– slope which corresponds to an "average" probe (setting of 5 μA probe rated current).

This makes it possible to start calibration with preset initial conditions.

The mode is to be used if analyzer calibration performance is doubtful.

2.6.8.1 Setting of zero hydrogen shifting

1 Switch off the analyzer.

2 Press the "**CAL**" button and holding it switch on the analyzer. On sound signal release the "**CAL**" button. The display shall show "***cd0.0***" message.

3 Press the "**ENTER**" button. The display shows "***donE***" for a short time and then "***cd0.0***" message. Press the "**MODE**" button to allow the analyzer enter the measurement mode. The display shall show indications in µg/dm^3 with "zero" shifting.

2.6.8.2 Setting of 5 µA rated current

1 Switch off the analyzer.

2 Press the "**CAL**" button and holding it switch on the analyzer. On sound signal release the "**CAL**" button. The display shall show "***cd0.0***" message. Press the "**CAL**" button once again. The display shall show "***cd5.0***" message.

3 Press the "**ENTER**" button. The display shows "***donE***" for a short time and then "***cd5.0***" message. Press the "**MODE**" button to allow the analyzer enter the measurement mode. The display shall show indications in µg/dm^3 corresponding to average analyzer slope.

On completion of the analyzer initial settings, proceed with 2.3.4.

3 MAINTENANCE

Analyzer maintenance comprises a regular hydrogen calibration and check of "zero" point in the range of measurement.

Analyzer hydrogen calibration is undertaken:

- after filling electrolyte on analyzer delivery;
- on a weekly basis;
- if analyzer workability is doubtful;
- after replacement of membrane assembly or teflon film; and
- after analyzer repair or after a long storage.

Check of "zero" point in the range of measurement is to be undertaken:

- after filling electrolyte on analyzer delivery;
- if analyzer workability is doubtful;
- after replacement of membrane assembly or teflon film; and
- after analyzer repair or after a long storage.

4 DELIVERY SET

4.1 Delivery set is shown in Table 4.1.

Table 4.1

Description	Quantity
1 Dissolved hydrogen analyzer (with hydrogen probe, cable length – 2 m)	1
2 Spare parts set: – probe spare parts; – tools and appliances kit.	1
3 Operation manual	1

APPENDIX A

(reference)

Solubility of hydrogen being in equilibrium with water vapor in distilled water according to temperature

P_{atm} = 101,325 kPa

Table A.1 in µg/dm^3

t °C	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
0	1922	1920	1918	1916	1914	1913	1911	1909	1907	1905
1	1904	1902	1900	1898	1896	1895	1893	1891	1889	1888
2	1886	1884	1882	1880	1879	1877	1875	1873	1872	1870
3	1868	1866	1865	1863	1861	1859	1857	1856	1854	1852
4	1851	1849	1847	1845	1844	1842	1840	1838	1837	1835
5	1833	1831	1830	1828	1826	1825	1823	1821	1819	1818
6	1816	1814	1813	1811	1809	1807	1806	1804	1802	1801
7	1799	1797	1796	1794	1792	1791	1789	1787	1785	1784
8	1782	1780	1779	1777	1775	1774	1772	1771	1769	1767
9	1766	1764	1762	1761	1759	1757	1756	1754	1752	1751
10	1749	1748	1746	1744	1743	1741	1739	1738	1736	1735
11	1733	1731	1730	1728	1727	1725	1723	1722	1720	1719
12	1717	1716	1714	1712	1711	1709	1708	1706	1705	1703
13	1701	1700	1698	1697	1695	1694	1692	1691	1689	1688
14	1686	1685	1683	1681	1680	1678	1677	1675	1674	1672
15	1671	1669	1668	1666	1665	1663	1662	1660	1659	1657
16	1656	1654	1653	1651	1650	1659	1647	1646	1644	1643
17	1641	1640	1638	1637	1635	1634	1633	1631	1630	1628
18	1627	1625	1624	1623	1621	1620	1618	1617	1615	1614
19	1613	1611	1610	1608	1607	1606	1604	1603	1601	1600
20	1599	1597	1596	1594	1593	1591	1590	1588	1587	1585
21	1584	1582	1581	1579	1578	1576	1575	1573	1572	1571
22	1569	1568	1566	1565	1563	1562	1561	1559	1558	1556
23	1555	1554	1552	1551	1550	1548	1547	1545	1544	1543
24	1541	1540	1539	1537	1536	1535	1533	1532	1531	1530
25	1528	1527	1526	1524	1523	1522	1521	1519	1518	1517
26	1515	1514	1513	1512	1511	1509	1508	1507	1506	1504
27	1503	1502	1501	1500	1498	1497	1496	1495	1494	1492
28	1491	1490	1489	1488	1486	1485	1484	1483	1482	1481
29	1480	1478	1477	1476	1475	1474	1473	1472	1470	1469
30	1468	1467	1466	1465	1464	1463	1462	1460	1459	1458
31	1457	1456	1455	1454	1453	1452	1451	1450	1449	1448
32	1446	1445	1444	1443	1442	1441	1440	1439	1438	1437
33	1436	1435	1434	1433	1432	1421	1420	1419	1418	1417
34	1426	1425	1424	1423	1422	1421	1420	1419	1418	1417
35	1416	1415	1414	1413	1412	1411	1410	1409	1408	1407
36	1406	1405	1404	1403	1402	1401	1400	1399	1398	1397
37	1396	1395	1394	1393	1392	1391	1390	1389	1388	1387
38	1386	1385	1384	1383	1382	1382	1381	1380	1379	1378
39	1377	1376	1375	1374	1373	1372	1371	1370	1369	1368

Table A.1 (continued)

t °C	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
40	1367	1366	1365	1364	1364	1363	1362	1361	1360	1359
41	1358	1357	1356	1355	1354	1353	1352	1351	1350	1349
42	1349	1348	1347	1346	1345	1344	1343	1342	1341	1340
43	1339	1338	1337	1336	1335	1334	1333	1333	1332	1331
44	1330	1329	1328	1327	1326	1325	1324	1323	1322	1321
45	1320	1319	1318	1317	1316	1316	1315	1314	1313	1312
46	1311	1310	1309	1308	1307	1306	1305	1304	1303	1302
47	1301	1300	1299	1298	1297	1296	1295	1294	1293	1292
48	1291	1290	1289	1288	1287	1286	1285	1284	1283	1282
49	1281	1280	1279	1278	1277	1276	1275	1274	1273	1272
50	1271	1270	1269	1268	1267	1266	1265	1264	1263	1262
51	1261	1260	1259	1258	1257	1256	1255	1254	1253	1252
52	1251	1250	1249	1247	1246	1245	1244	1243	1242	1241
53	1240	1239	1238	1237	1236	1234	1233	1232	1231	1230
54	1229	1228	1227	1226	1224	1223	1222	1221	1220	1219
55	1218	1216	1215	1214	1213	1212	1211	1210	1208	1207
56	1206	1205	1204	1202	1201	1200	1199	1198	1196	1195
57	1194	1193	1192	1190	1189	1188	1187	1185	1184	1183
58	1182	1180	1179	1178	1177	1175	1174	1173	1172	1170
59	1169	1168	1166	1165	1164	1162	1161	1160	1158	1157
60	1156	1154	1153	1152	1150	1149	1148	1146	1145	1144
61	1142	1141	1139	1138	1137	1135	1134	1132	1131	1130
62	1128	1127	1125	1124	1122	1121	1119	1118	1117	1115
63	1114	1112	1111	1109	1108	1106	1105	1103	1102	1100
64	1099	1097	1095	1094	1092	1091	1089	1088	1086	1085
65	1083	1081	1080	1078	1077	1075	1073	1072	1070	1068
66	1067	1065	1063	1062	1060	1058	1057	1055	1053	1052
67	1050	1048	1047	1045	1043	1041	1040	1038	1036	1034
68	1033	1031	1029	1027	1025	1024	1022	1020	1018	1016
69	1015	1013	1011	1009	1007	1005	1003	1001	1000	998
70	996	994	992	990	988	986	984	982	980	978