



EXPERTS IN WATER CHEMISTRY SINCE 1903



9091C Dissolved Hydrogen Analyzer User Manual

Revision 2.04



WALTRON CUSTOMER COMMITMENT

This instruction manual is a technical guide to aid the customer in the set-up, operation, and maintenance of their new Waltron measuring system. Waltron provides continuous product improvement and reserves the right to make any modifications to the information contained herein without notice.

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Technical questions concerning this product should be addressed to:

Waltron Technical Service Department
Flemington, New Jersey
Phone: (908)-534-5100
Fax: (908)-534-5546
www.waltron.net

Please be ready to provide the following information:

- Date analyzer was purchased
- Analyzer model and serial number
- Recent maintenance history
- Calibration slope values and detailed description of problem

Waltron's technical expertise and extensive experience provides personalized solutions to the water quality industry. It is Waltron's commitment to provide the customer with timely and accurate technical service and support.

Waltron fully expects the customer to be satisfied with the quality, performance, and cost of this product.

If there are any questions or concerns regarding this product, please feel free to contact Waltron at (908)-534-5100.

Thank you for choosing Waltron!

Please note the Waltron mailing and shipping address:

Waltron Bull & Roberts, LLC
25 Minneakoning Road, Suite 101
Flemington, NJ 08822



Safety:

Please observe proper safety and handling precautions when installing, operating, maintaining, and servicing this product. The following should be noted and adhered to:

- √ Read and understand manual before working with analyzer.
- √ Pay special attention to warning labels on enclosures, containers, packages and chemicals.
- √ Only qualified personnel should be involved in the installation, operation, and servicing of the analyzer.
- √ Follow safety precautions when operating analyzer in conditions of high pressure and/or temperature.
- √ Keep analyzer chemicals away from heat and extreme temperatures. Reagent powders must be kept dry.
- √ Follow all regulations and warning labels when disposing of chemicals. Do not mix chemicals.

To obtain analyzer safety information or Safety Data Sheets (SDS), please contact Waltron or visit the website at www.waltron.net .



Warranty Agreement

If, within one year from the date of shipment, the customer experiences any equipment defects or is not satisfied with the analyzer manufacturing, Waltron will repair, or at its option, replace any defective part(s) free of charge. This warranty requires that the defective part(s) be returned to Waltron with shipping charges prepaid.

At Waltron discretion, a Technical Service Specialist may be sent out to repair or replace the defective part(s) on location. Traveling time and expenses of the Technical Service Specialist is at the customer's expense.

Equipment sent to Waltron must be appropriately packaged and the following information must be provided prior to returning to Waltron:

- The Return Authorization (RA) number assigned to the customer by the Waltron Technical Service Department
- Customer name, address and department
- Name and telephone number of the individual responsible for returning items for repair
- Brief problem description

Ship to Waltron service center:

Waltron Bull & Roberts, LLC
25 Minneakoning Road, Suite 101
Flemington, NJ 08822

The Waltron Warranty Agreement:

- Covers expendable sensors for one month after shipment and reusable electrodes for six months after shipment.
- Does not apply to damages occurred during shipping.
- Warranty will be nullified if goods have been used for purposes other than those for which they are intended or if any seal has been removed, broken or tampered with or if the Waltron trademark or serial number has been removed, defaced, or altered.
- Does not cover expendable supply items such as reagents, tubing and electrolytes.
- Does not cover misuse or mistreatment by the user.
- Does not cover previous repair or alteration by unauthorized individuals.

Waltron does not assume responsibility for contingent liability through alleged failure or failures of products or product accessories.



Checklist of Materials

In order to ensure customer satisfaction, Waltron does its best to provide adequate and timely packaging and shipping services. Please perform the following after receiving a shipment:

- √ Inspect all shipping containers upon receipt and record any visible damage. If there are any outward signs of damage, please retain all containers and packages for inspection by carrier. Please retain all packing material so that it can be used for future moving and shipping needs.

- √ Check all items received against those on the packing list. Chemicals are usually shipped in a separate package and will be itemized accordingly.

- √ Verify that the number of packages received agrees with the packing list and shipping papers.

- √ Notify both Waltron and the carrier if any problems occur.

Important Notice:

- All analyzers are inspected and tested prior to shipment.
- In normal use, the unit should require only minor maintenance and should operate correctly and without fault over a long period of time.
- Please note that if electronic components need to be replaced, it may be necessary to adjust and/or calibrate the analyzer.
- Failure to carry out correct maintenance procedures may result in inaccurate analyzer readings.



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1 INTRODUCTION

1.1 AREAS OF APPLICATION

The Waltron 9091C Dissolved Hydrogen Analyzer is used for the automatic, continuous measurement and control of hydrogen concentration in aqueous solutions. Examples of the applications of the unit include the determination of residual hydrogen concentration in boiler feedwater in power plants, the monitoring of hydrogen concentration in the primary loop of a nuclear power plant, the monitoring of hydrogen concentration during the denitrification (removal of nitrate NO_3) of drinking water, or the indication of the hydrogen concentration during the catalytic reduction of oxygen on noble metal surfaces in water treatment plants etc. The measuring range is between some $\mu\text{g/l}$ (trace areas) up to saturation level mg/l range.

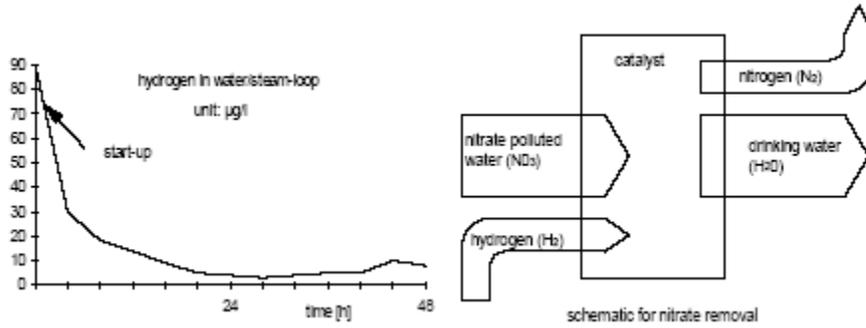
The hydrogen in a water/steam loop of a power plant is mainly produced by the reaction between iron and water or steam. Thus the hydrogen concentration gives quantitative and qualitative evidence of the rate of corrosion and the buildup or deterioration of the protective oxide layer inside pipes and boiler tubes. The hydrogen concentration ranges between a few $\mu\text{g/l}$ (normal operating conditions) and several hundred $\mu\text{g/l}$ during the start-up.

In the primary loop of a nuclear power plant the hydrogen should reduce or mitigate the oxygen which is produced by radiolysis. As a result the electro-chemical corrosion potential (ECP) of the tube material is reduced. In nuclear power plant applications, hydrogen is added to scavenge oxygen or recombine with the oxygen that is produced by radiolysis. In the pressurized water reactor (PWR) primary loop, hydrogen is measured in the range 25-50cc/kg. Hydrogen in boiling water reactors (BWR) feedwater and reactor coolant is measured in ppm's. Hydrogen in feedwater is typically 0-2ppm and hydrogen in reactor coolant water will vary between 0.1-0.5ppm.

The dissolved hydrogen analyzer can also be used in drinking water applications for the denitrification of drinking water. The denitrification is based on the process in which hydrogen is added to nitrate polluted water. The contaminated water flows through a solid bed reactor filled with a noble metal catalyst. In result, water and nitrogen are produced.

The catalytic reduction of oxygen is based on the reaction between dissolved oxygen in water and hydrogen in the presence of a noble metal surface (oxyhydrogen gas reaction). Typical applications for oxygen free water are in breweries, the beverage industry and in the preparation of boiler feedwater in power plants.

It is important for the correct operation of various processes to dose the exact amount of hydrogen.



1.2 DESCRIPTION OF DEVICE

The Waltron 9091C Dissolved Hydrogen Analyzer measures reliably and quickly the hydrogen concentration in aqueous media from the trace areas (<10,0 µg/l) up to saturation concentrations of higher than 1600 µg/l under normal conditions.

The Waltron 9091C Dissolved Hydrogen Analyzer can be divided into two primary function parts. The first is the electronic component which consists of the display and input elements as well as the measuring data collection and data processing elements.

The second is the analytical component which is composed of the inlet valve, filter device and salt cell which serve the sample and probe preparation. The measuring device is situated together with the calibration cell, the measuring cell and the flow measuring device. The measuring cell is joined with the temperature sensor and the reference cell with its reference electrode.

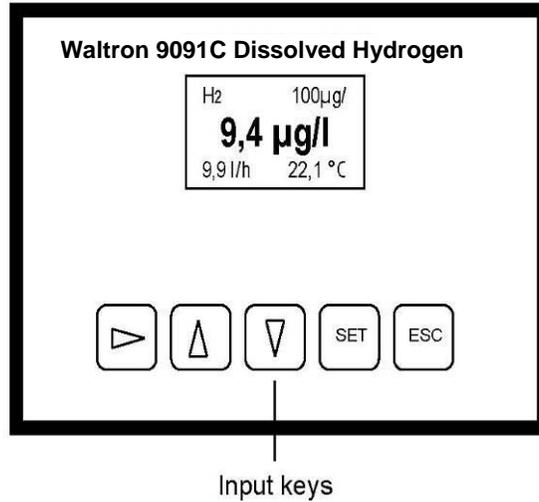
For the measurement of the dissolved hydrogen (molecular hydrogen H₂) the sample flows through the components of the device which relate to the sample and probe preparation and through the calibration cell into the coaxial measurement cell (grooved measurement cell) passing by the platinum measuring electrode. The hydrogen measurement reaction takes place due to the potential for hydrogen oxidation in the boundary layer (interphase) between the electrode surface and sample medium.

The analyzer features automatic in-line calibration. A stainless steel electrode is used in the calibration process. This electrode produces a known amount of H₂ by electrolysis.



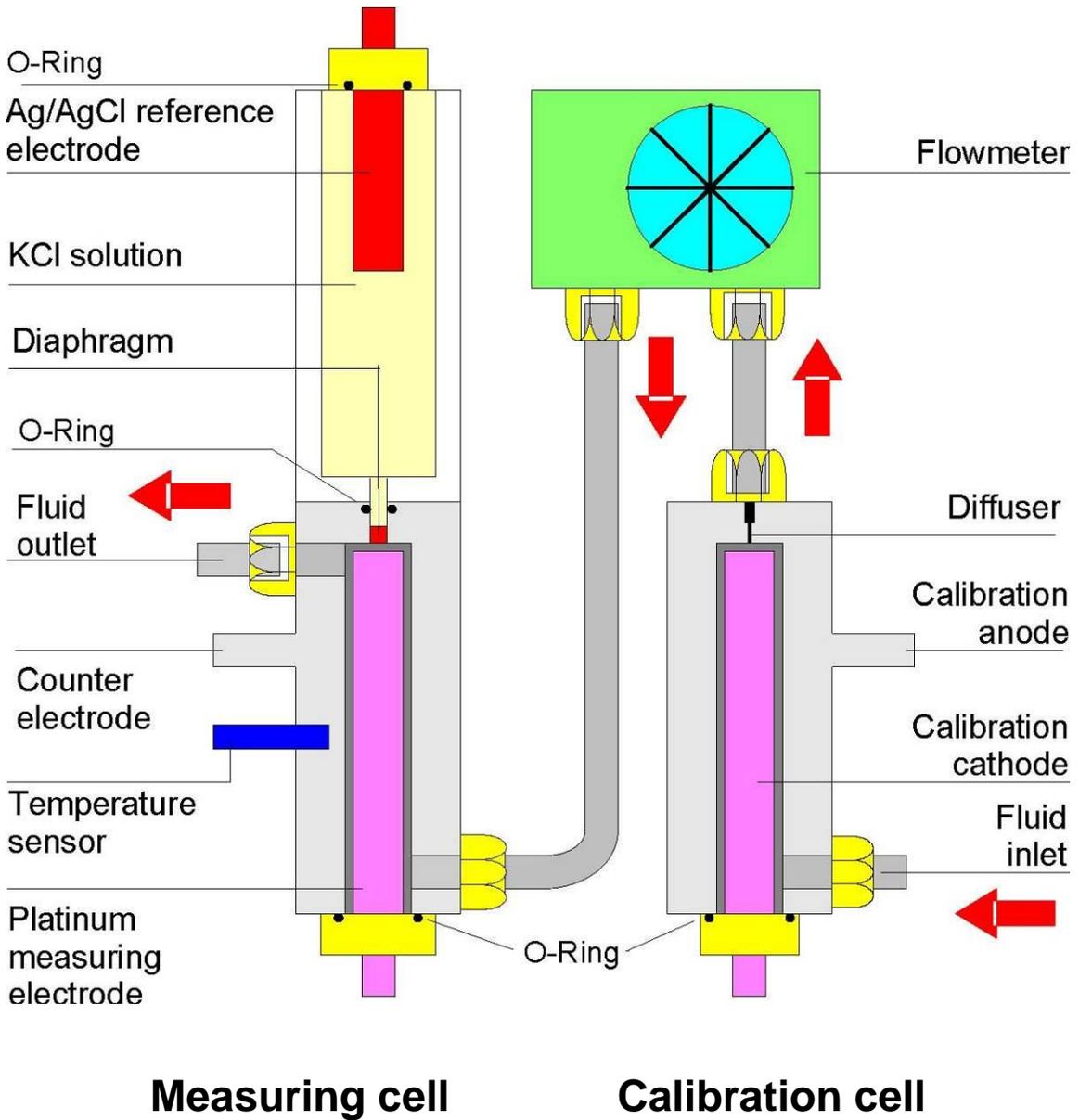
The fully automatic calibration is activated with the push of a single button. As no other external calibration devices or procedures are required, it is not necessary to shut down the operation during the calibration. This sturdy and reliable measurement system makes it possible to operate very accurately, even under extremely harsh operating conditions.

Front view



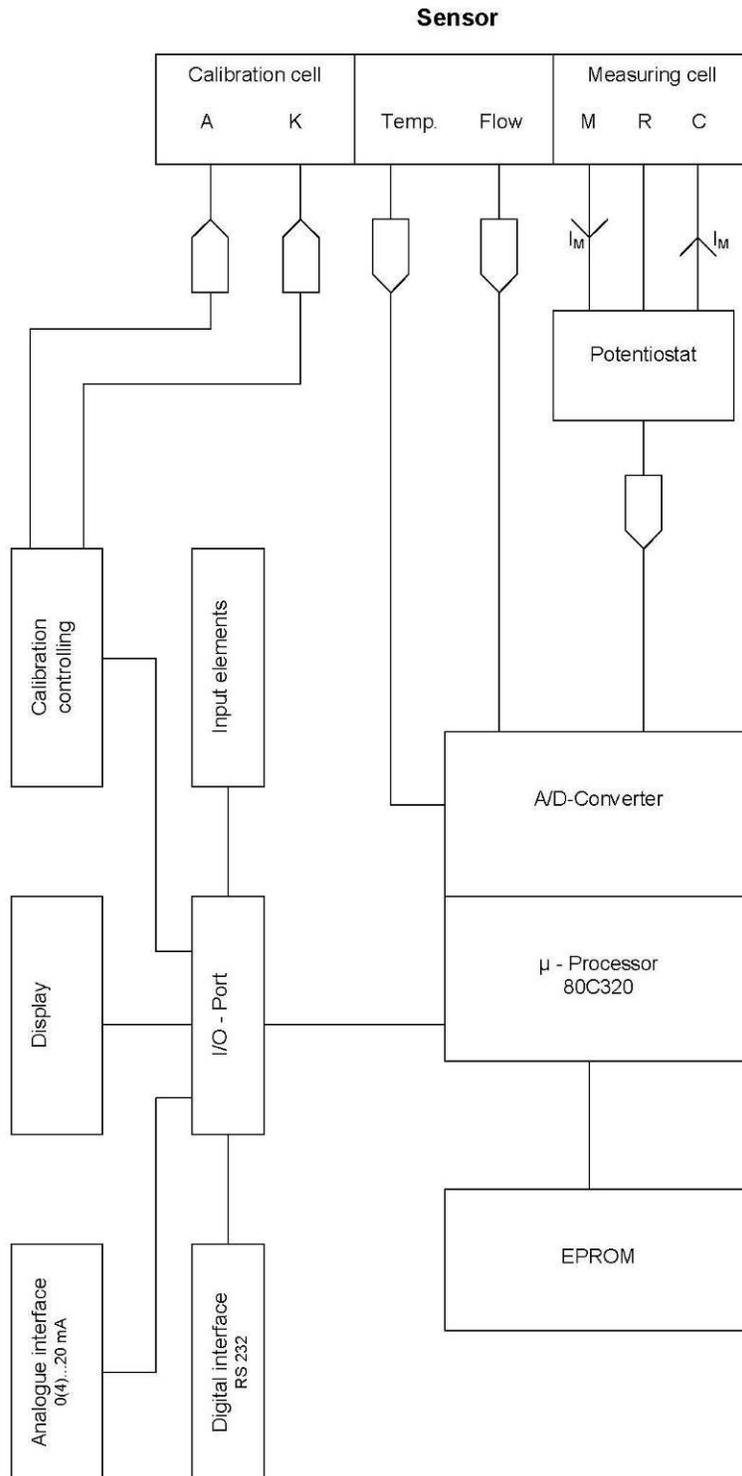


1.3 THE MEASURING SYSTEM





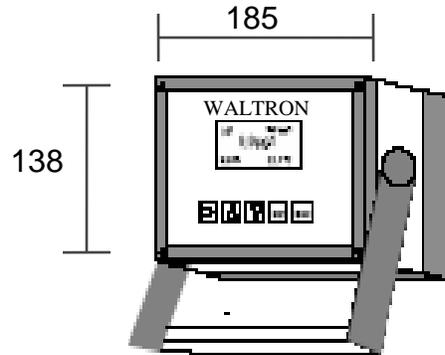
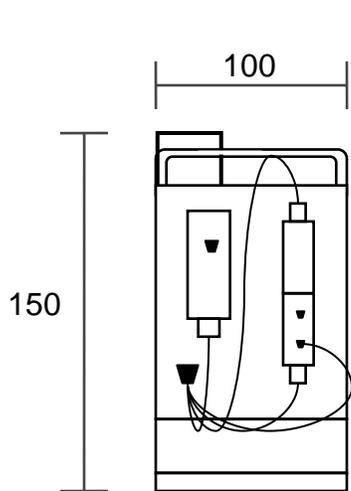
1.4 BLOCK DIAGRAM OF SENSOR SYSTEM



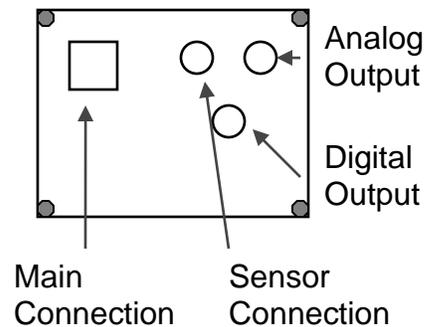


2 INSTALLATION

2.1 *DELIVERED PARTS*



Plug Arrangement Rear



Color Code Analog Output

White	→	0/4.....20 mA +
Brown	→	0/4.....20 mA -
Green	→	Limit (low)
Yellow	→	Common
Gray	→	Limit (high)

2.2 *ACCESSORIES*

1. 100 ml KCl-solution
2. 1 kg granular marble
3. 1 test tube brush
4. 1 cleaning material
5. 1 power cable
6. 1 recorder cable
7. 1 pocket (option)
8. 1 sample line (option)
9. 1 software for digital measuring (option)



2.3 TECHNICAL DATA

Measurement principle:.....	microprocessor based, potentiostatic three electrode system
Calibration:.....	build-in, single button operation
Auto-calibration:.....	option
Measuring range:	
Measuring group I:.....	0,0.....500,0 μ g/l range selectable between 20....500 μ g/l
Measuring group II:.....	0,00.....20,00 mg/l range selectable between 4....20 mg/l
Auto-changing-range:.....	option
Analog output:.....	0(4).....20 mA; shunt max. 500 Ohm
Digital output:.....	serial interface RS 232
Data logging:.....	option
Limit:.....	power relay
Alarming:.....	power relay; Flow and Calibration
Measuring electrode:.....	platinum
Counter electrode:.....	stainless steel 1.4571 (314)
Reference Electrode:.....	Ag/AgCl in saturated KCl-solution
Calibration electrode:.....	stainless steel 1.4571 (314)
Response time t90:.....	30 sec
Probe conductivity:.....	$\geq 10 \mu$ S/cm; (if conductivity is less a salt cell is required)
Ambient temperature:.....	0....+55 °C
Sample temperature:.....	0....+60 °C
Sample pressure:.....	≤ 8 bar (116 psi)
Sample flow:.....	5 l/h15 l/h
Sample fittings:.....	tube fittings for tube \varnothing 6 mm
Error limits:.....	$\pm 3\%$
Protection class:.....	IP 54
Color:.....	basic parts RAL 7035; front and rear parts RAL 7024
Voltage:.....	100...240 VAC, 50/60 Hz
Power consumption:.....	10 VA

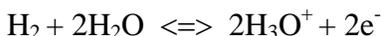


3 OPERATING PROCEDURES

3.1 PRINCIPLE OF MEASUREMENT

The new patented 9091C Dissolved Hydrogen Analyzer electrochemical hydrogen sensor functions in a potentiostatic mode with an open three-electrode system. The measuring electrode is made of solid titanium, coated with platinum. The counter electrode is made of stainless steel and the reference electrode of silver/silverchloride. The use of an open measuring electrode means, that there is a direct contact between the sample and the measuring electrode. A membrane is not required.

For the measurement of dissolved molecular hydrogen (H₂), the fluid medium flows into the coaxial measurement cell between the platinum measuring electrode and counter electrode. If the measuring electrode has reached the correct potential, hydrogen is oxidized in the interphase boundary layer. The electrochemical reaction may be represented qualitatively as:



The characteristic potential for this reaction- the potential at which hydrogen oxidation occur in preference to other competing reactions- is measured by holding the potential of the measuring electrode steady with a potentiostat and comparing the value with the value of the reference electrode. Under these conditions the rate of the electrochemical reaction is limited by the rate of diffusion of hydrogen molecules to the electrode. As long as we remain within this 'diffusion-limiting-current' area, the electrochemical cell delivers an electric current, whose size depends linearly on the concentration of dissolved hydrogen. This current, which is measured by the instrument's electronic processing unit, is known as the diffusion-limiting current, I_{diff} :

$$c_{\text{H}_2} = k \cdot I_{\text{diff}}$$

This diffusion-limiting current depends mainly on the mass transport of the electrochemically active components, and hence on the hydrodynamics in the measuring cell. It therefore changes significantly with the temperature and flow rate through the measuring cell. In practice the flow rate and temperature vary over wide ranges, but the instrument corrects for these changes using empirical correlations.

A second important influence on the measurement is the condition of the electrodes. In particular, the adsorptive and oxidative state of the electrode surface layers. This is independent of the flow conditions. It depends instead on the voltage between the electrode and the electrolyte, and on the material of which the electrode is made.

Appearing flow-and temperature effects of the measuring signal are recorded with the aid of the installed flow meter and the temperature sensor and can be compensated, if it is necessary.



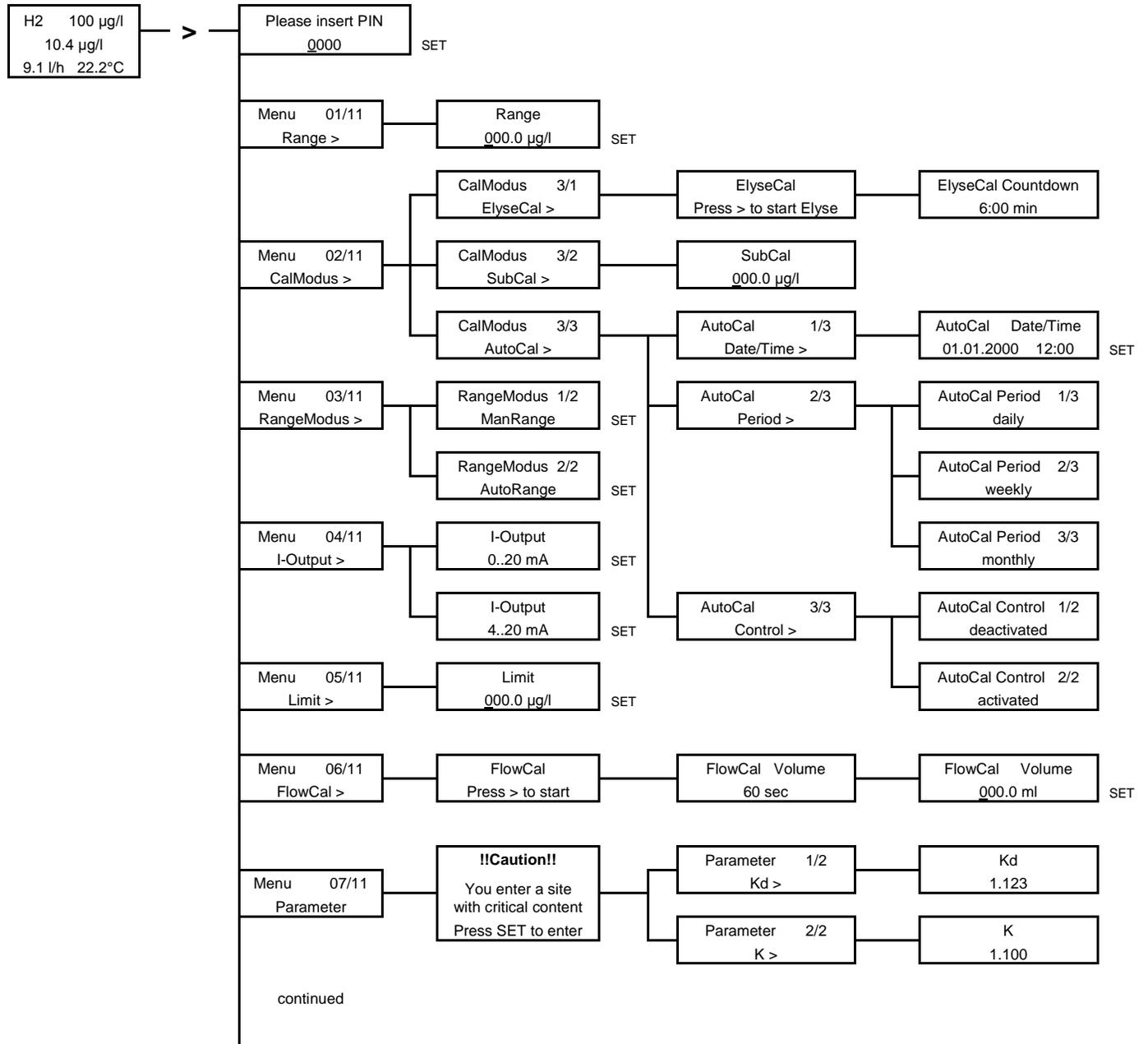
3.2 CALIBRATION VIA ELECTROLYSIS

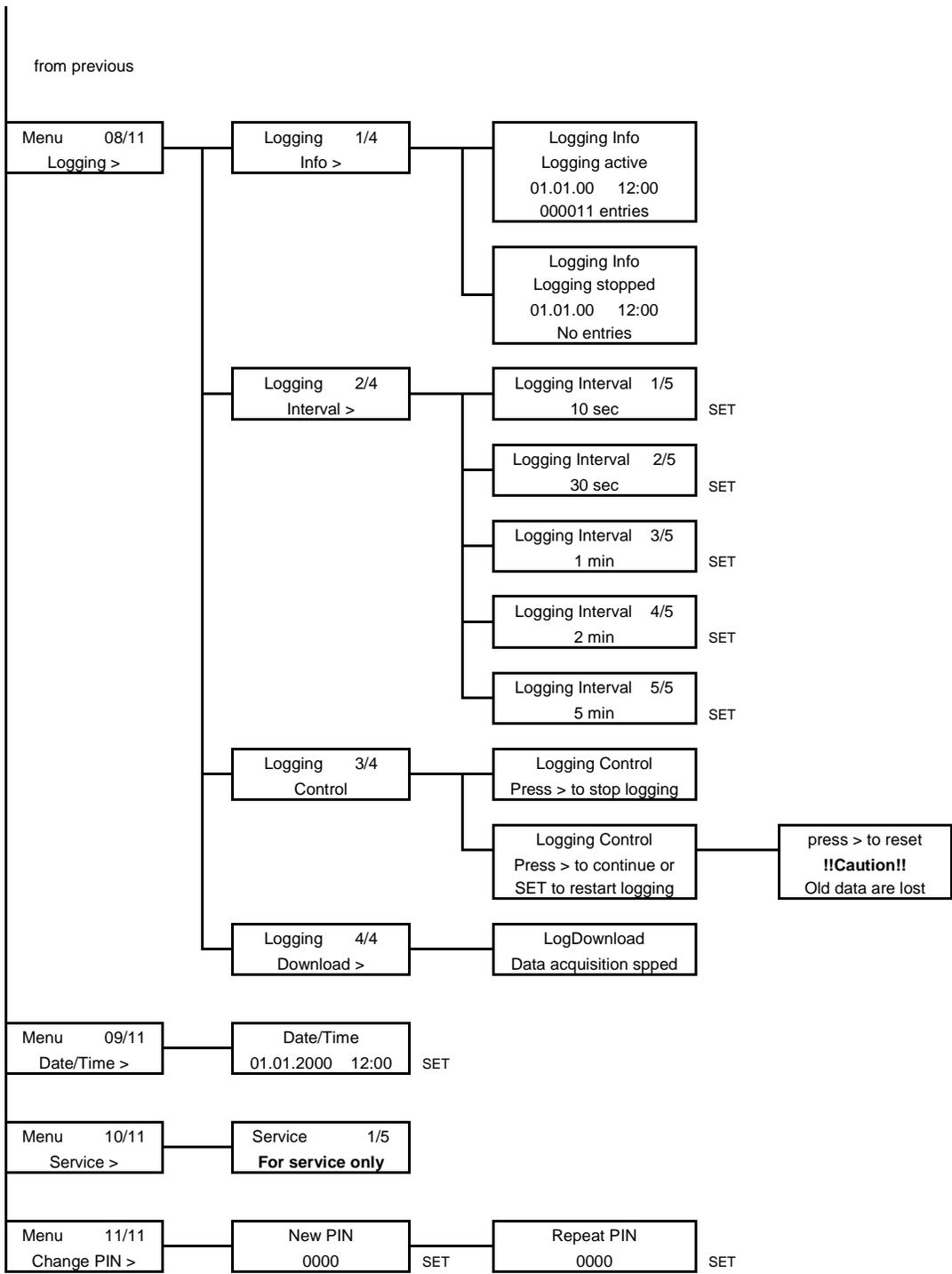
The calibration cell contains two stainless steel electrodes, between which water is dissociated by a constant, controlled, current. According to Faraday's law the rate of hydrogen production in the calibration cell is directly proportional to the current. A constant electric current of 13.4 mA, for example, produces an increase in the hydrogen concentration of 50 $\mu\text{g/l}$ at a flow rate of 10 l/h. This rise of the output current of the measuring cell, allows the processing unit to calibrate the system automatically. It is important to keep the operation parameters nearly constant while running the calibration process. Especially the basic value of the measured component must not change during this period.



4 START-UP

4.1 MENU STRUCTURE







4.2 PREPARING TO MEASURE

Before you start measuring you may perform the following procedures.

1. The electrical power supply has to be connected according to the local power authority guidelines with the terminal box, with which the connectors to the 0(4)...20 mA analog output (see the circuit diagram on the cover plate) should be combined.
2. The reference cell must be filled with KCl solution. The reference cell must be removed from the measuring cell by unscrewing the reference cell. Take out the reference electrode and fill up the KCl solution to the level of about the beginning of the external threading. Make sure that no air bubbles develop near the diaphragm. Tap the cell gently if necessary. Remove any surplus of the KCl solution and reinsert the reference electrode. Make sure that the installation of the O-ring is correct.
3. If the probe has a conductivity of $\leq 10 \mu\text{S}/\text{cm}$, the salt cell must be filled with granular marble (calcium carbonate CaCO_3). To do this unscrew the cover and pour the granular marble carefully into the large cavity.
4. The probe tube is connected with tube fittings to the inlet valve. The outlet line is placed at the measuring cell exit with a hose having a 5 mm inside diameter.

4.3 CHOICE OF OPERATING PARAMETERS

Before starting measurement, set your operating parameters. With the keys at the front site you can move through the menu. To enter the menu, press key \triangleright and insert your code number (**Pre setting: 0000; see page 17**). With the keys ∇ and \triangle you move one step in the vertical direction up or down and also increase or decrease numbers. With the key SET you confirm chosen parameters, with the key ESC you jump back one step in the horizontal direction with **no effect**.

If a > sign is displayed, you can go with the key \triangleright right ahead in a sub-level.

The menu consists of eleven levels. You find your present position on the right top of the display.

1st Menu level *Range* > shows range settings. The value is corresponding to the maximum analog output current of 20 mA. Press key \triangleright and make your choice by setting digit for digit and press the adjacent key SET. Your range is now selected and the indication jumps one step back in the *Menu*.

2nd Menu level shows the calibration settings named *CalModus* >. Press key \triangleright to jump to the sub-level *CalModus* > which consists of three sub-levels called: *ElyseCal* >, *SubCal*



> and *AutoCal* >. (**option only**). With the key ▷ you can go ahead. With the key ▷ you will release the electrolytic calibration if you start from position *ElyseCal* >. From *SubCal* > you are setting the present hydrogen concentration digit for digit, finally push key SET.

3rd *Menu* level *Range Modus* > gives you the choice between *ManRange* (range which is setting in 1st level only) and *AutoRange* (automatic changing range which has double value of *ManRange* >)(Option only).

4th *Menu* level *I-Output* > sets the analog output current 0(4)...20 mA.

5th *Menu* level *Limit* > allows limit settings. There exists a switch hysteresis of 10% of the chosen limit.

6th *Menu* level *FlowCal* > enables flow meter calibration (see *Section 7*).

7th *Menu* level *Parameter* > shows operation parameters Kd (flow) and k (calibration).

Attention: Only reading parameter.

8th *Menu* level *Logging* > is the data logging function (Option only).

9th *Menu* level *Date/Time* > allows date settings.

10th *Menu* level *Service* > **is for service only.**

11th *Menu* level *ChangePIN* > gives you the possibility to change your PIN number.

Attention: lost PIN numbers can only activated by the manufactures.



5 MEASURING MODE

5.1 OPERATING CONDITIONS

For an accurate measurement of hydrogen the probe's environment must fulfill certain conditions regarding conductivity, flow, temperature and pressure.

- The probe conductivity must be $\geq 10 \mu\text{S}/\text{cm}$; otherwise the salt cell must be refilled (see also *Section 4.2*).
- The flowrate has to be between 3 l/h and 18 l/h (The optimal flowrate is about 10 l/h).
- The temperature of the sample should range between 1 °C and 60 °C.
- The pressure to which the probe should be subjected at least 400 mbar and may not exceed 8 bar. Operating conditions outside this range require either a probe pump or a pressure reducing valve.

Connect the probe line on the fitting tube of the inlet valve. Adjust the flow very carefully and slowly. You have to make sure that no sudden pressure push occurs and you should verify that all air bubbles in the tube system rinse out. To help the bubbles to rinse out, open and close quickly the inlet valve several times.

Attention: Make sure that the plugs stay dry.

5.2 ANALYZER CALIBRATION

After the hydrogen analyzer is installed and the medium flows through the analyzer the calibration can be performed. The first calibration should take place after letting the sample flow, for approximately 30 minutes. During the calibration the operation conditions should not change.

1) You are in the mode for hydrogen measurement and have pressed key \triangleright and insert your PIN number. Select *ElyseCal* > in the menu (only for hydrogen or oxygen measurement). You start the calibration procedure with key \triangleright . The display indicates:

ElyseCal Countdown 6:00 min

Thereby a countdown runs; beginning at 6:00. After the calibration time of 6 minutes is over, the calibration mode changes automatically to the measuring mode and the microprocessor calculates the current sensitivity of the measuring electrode and gives the electrode a k-Factor. This k-Factor is displayed in the menu *Parameter* > (7th level).



2) If you selected the calibration mode *SubstCal* > in the *CalModus* :
You start the calibration procedure by pressing key ▷ (suitable for Hydrogen measuring).
The display changes to:

SubCal 000.0 µg/l

The setting of the present hydrogen concentration is performed digit for digit. If the value is correct, press key SET. Your calibration value is stored and the instrument determines the current sensitivity of the measuring electrode by the modern microprocessor. The indication jumps one step back in the menu. The **k-Factor** value can be displayed in the same way described above. To reach the measuring mode, press the key ESC twice. If the shown value is beneath 10 % of the value determined through the calibration, which followed after a cleaning procedure, a new cleaning of the electrode is necessary.

6 MAINTENANCE

6.1 *CLEANING OF ELECTRODES*

Reference electrode: The surface of the reference electrode is made of silver chloride (AgCl), by a galvanic process, and must not be cleaned.

Measuring electrode: The measuring electrode has to be cleaned (activated) if the sensitivity (k-Factor) of the measuring electrode is beneath 10 % of the determined value, calculated after that calibration, which followed a cleaning. As discussed above, the k-Factor can be looked up in the menu Parameter > in the 7th level.

Before you remove the measuring electrode, it is necessary to empty the analyzer (of probe medium). Then remove the plug (M) from the measuring electrode and unscrew the thrust screw of the electrode. Take the electrode slowly out of the cell. You should be careful not to drop the measuring electrode and not to hit it against the cell wall. Clean the surface with a mild detergent, by rubbing gently the electrode between your middle finger, index finger and thumb. During the cleaning, do not press too hard the electrode. This procedure should not last longer than 2 minutes (the Pt-layer is only several µ m thick). Rinse well with water and do not touch the surface of the electrode with your fingers thereafter.

Occasionally a wiping of the electrode surface with a clean tissue is sufficient. Thereby you reduce the later start up time until steady state condition.

Calibration electrode: The cleaning cycle and cleaning procedure for the calibration electrode are the same as described above for the measuring electrode.



6.2 CLEANING OF CELLS

The cleaning cycle of both the counter electrodes of the measuring and calibration cells is the same as those of the measuring electrode and calibration electrode. To clean the counter electrodes, you close the inlet valve and remove the plug connectors (A), (K) for the calibration cell, as well as (M), (C) and (R) for the measuring cell. Disconnect the pipe between the calibration cell and the flowmeter. Disassemble the calibration cell from the square mounting by detaching the fitting (screw width SW 12 mm). Remove the calibration electrode (see also the instruction for cleaning electrodes above). Clean the interior with a mild detergent and a thin test tube brush (see also "Spare parts"). Examine, if the nozzle in the top part (diameter \varnothing 0.8 mm) is free. After the cleaning you have to rinse the cell well with water. The dismantling of the measuring cell from the square mounting should be performed in the same way as the calibration cell. Remove the outlet pipe and unscrew the reference cell from the measuring cell (see also *Section 4.2*). Take the electrode slowly out of the cell (see also *Section 6.1*). The cleaning procedure thereafter is the same, as described for the calibration cell.

When reassembling, make sure that all parts are dry.

7 TROUBLESHOOTING

- If the values of the flow measurements seem incorrect, a calibration of the flow meter should be performed. The procedure has to be as follows:
 1. Adjust the flow to approximately 10 l/h and have available a 250 ml graduated cylinder.
 2. Choose the *FlowCal* > level and press the key \triangleright and follow the displayed instructions.
 3. If the countdown runs beginning at 60 sec collect the flow during the 60 second period in the graduated cylinder. When the countdown is completed, stop collecting the flow.
 4. The amount (in ml) of sample, collected in the cylinder during the 60 seconds, should be entered digit by digit with the keys Δ , ∇ , and \triangleright . If the value is correct, press key SET. Press key ESC to go back to the measuring mode.
- If you are obtaining unlikely results after a hydrogen calibration, check the following points:
 1. Verify the interior of the calibration cell for short-circuits between the calibration electrode and the counter electrode.
 2. Remove the (A) and (K) plugs from the calibration cell and plug in an instrument to measure a electric current. If you trigger a calibration with the "Cal." key, a current of 13.4 mA must be observed.



- If you observe unsteady or unlikely results during the measurements, check if the plug connectors are dry and make sure that the reference electrode is completely immersed into the KCl-solution.
- During longer periods, where no measurement takes place, (for example longer than 3 weeks) it is advisable to clean the analyzer. There should not remain any probe material or KCl solution within the analyzer. In case of shorter breaks, parts can stay in wet surrounding. It will reduce the start up time and the time until steady state condition.

8 SPARE PARTS AND ACCESSORIES

1 Set of O-rings (TA).....	LP	100.01
100 ml of KCl-solution.....	LP	100.02
1 kg of granular marble.....	LP	100.03
1 Reference electrode.....	LP	100.04
1 Measuring electrode (Au/N ₂ H ₄).....	LP	100.05
1 Test tube brush.....	LP	100.06
1 Stck. reference cell.....	LP	100.07
1 Stck. cleaning material.....	LP	100.08
1 Measuring electrode (Ag/O ₂).....	LP	100.09
1 Measuring electrode (Pt/H ₂).....	LP	100.10
1 Salting cell (TA).....	LP	100.11
1 Salting cell (TI).....	LP	100.12
1 Sieve.....	LP	100.13
1 Set of O-rings (TI).....	LP	100.14
1 Flow meter (TA).....	LP	100.15
1 Flow meter (TI).....	LP	100.16
1 Electrode Holder.....	LP	100.17

TA parts for wall mounted instrument

TI parts for portable instrument