Operating Instructions

for the

PLUGSYS®-Module

pH Measurement Modul pHMM Typ 694

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1. Introduction, manufacturer's details

These Operating Instructions describe the operation and use of the **pHMM** Module Type 694. It is part of the equipment and should be kept close to it.

All the information in these Instructions has been drawn up after careful examination but does not represent a warranty of product properties. Alterations in line with technical progress are reserved.

This PLUGSYS module is manufactured by:

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Trademark

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2. Safety note

Important: This equipment is not suitable for operation in hazardous areas and/or in a flammable atmosphere.

The equipment is not approved for measurement on humans!

3. General description, applications

The **pHMM** Module Type 694 is a module for the HSE PLUGSYS Measuring System and serves to measure pH using glass pH electrodes. The main application is in the continuous recording of the pH in biological solutions such as e.g. the perfusate from isolated organs and tissues. Using a special pH electrode mounted in a flow-through chamber it is possible to make continuous measurements over hours or days. Changes or fluctuations in pH can be recorded continuously. In order to avoid interaction where several electrochemical parameters (such as pO_2, pCO_2, Na^+, K^+ or Ca^{++}) are being measured continuously, this module incorporates an input with isolating amplifier. This also avoid possible hum interference. The input circuit is isolated from the output circuit and the housing through the isolating amplifier.

The pH is indicated on a 3 1/2 digit LED display. It is possible to indicate either the signal supplied by the electrode in mV, or the pH calculated from it.

The pH signal is available as analogue voltage at a BNC socket on the front panel and also internally on the PLUGSYS system bus for recording.

A simulating device which can simulate two fixed pH values is provided for calibrating a recorder or a computer system.

For using the pHMM module it has to be installed in a PLUGSYS housing Series 600.

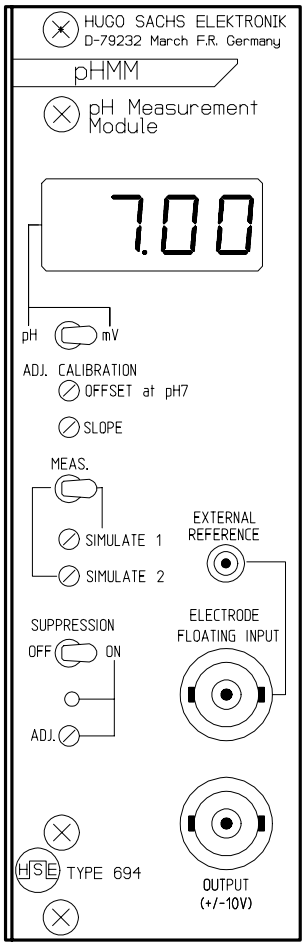


Fig. 1 Front panel

4. Installing the module in a housing

(If the module has been supplied already installed you can omit Section 4 and continue with Section 5. If you have received the module as a separate unit you should continue here.)

Before you can use the **pHMM** module it has to be installed in a suitable HSE PLUGSYS housing Series 600 (April. 96: 601 to 607). If the module is supplied as part of a completely installed PLUGSYS measuring system the work described below has already been carried out and the selected signal paths have been entered in the bus diagram.

Before the module is installed in a housing the connections of the module to the bus lines have to be determined by plugging in links as described in the next section (Section 4.1).

Do not forget to enter the selected connections in the bus diagram (in the white Operating Manual folder under Section 1).

Brief procedure (for full details see the Operating Manual of the housing):

- Pull out the mains plug on the housing.
- Remove the blank panel at the housing slot position intended for the **pHMM** module.
- Prepare module according to Section 4.1 (set lines and links).
- Insert the **pHMM** module, note the guide rails.
- Push the module firmly into the bus connector.
- Screw on the front panel.
- Connect up the pH electrode.
- Reconnect the mains plug to the housing.
- Switch on the housing.

4.1 Internal instrument settings, links

Warning: the **pHMM** module must be protected against electrostatic discharges while it is outside the housing! The **pHMM** module contains highly sensitive MOS components which can be damaged or destroyed by electrostatic discharges. If you dismantle the module or if you carry out any operations on the dismantled module you must ensure potential equilibration before touching any part of the printed circuit (by touching some grounded metal part, e.g. water tap, central heating radiator. grounded housing, PLUGSYS housing or similar).

Before you install the **pHMM** module into the PLUGSYS housing it is necessary to set a link on the circuit board in order that the output signal is linked to the appropriate or required bus line. The module can only be used in conjunction with the complete system if the bus lines have been connected up correctly.

Do not forget to enter the selected signal assignment in the bus diagram for the PLUGSYS housing (the bus diagram is filed in the Operating Manual folder under Section 1).

If the module is supplied as part of a completely installed PLUGSYS measuring system, the operations described below have already been completed and the selected signal paths have been entered in the bus diagram.

The location of the link is shown in the illustration below. The following linkages can be set:

- Signal output AV_PH to transfer the pH to the PLUGSYS bus system; suppression can be switched on.
- Signal output AV_MV to transfer the mV value to the PLUGSYS bus system
- Signal output AV_MV_SU to transfer the mV value or the pH to the PLUGSYS bus system, selected by switch

4.11 Signal output AV_PH to transfer pH to the PLUGSYS bus

Plugging the link AV_PH transfers the analogue signal corresponding to pH to the PLUGSYS bus. The link must be plugged on to the appropriate channel (AV1 - AV16). The signal is transmitted through the PLUGSYS bus to a recorder or to a data capture system. **See Fig. 2 C**.

Please note:
This signal is influenced by the **"SUPPRESSION"** switch. If the **SUPPRESSION** switch is set to **OFF** then 1 Volt = 1 pH. This means that 7 V is produced at this output for pH 7, pH 4 corresponds to 4 V. This leads to problems when using a recorder since its pen position control can not be adjusted over such a large range. This problem is overcome by using the **SUPPRESSION** function. If the **SUPPRESSION** switch is set to **ON** it is possible to shift the output voltage range. For example, pH 7 can be set to 0 Volt. This allows the range pH 7 to pH 8 to be shown enlarged on the recorder (pH 7.2 to 7.5 is the normal physiological range, for example).

Note:
When selecting the bus line (AV1...16) be sure to use a free line and check this in the bus diagram. If there is no appropriate information in the bus diagram you can determine the bus line assignment only by removing all the modules and determining the signal paths selected on them using the corresponding operating instructions.

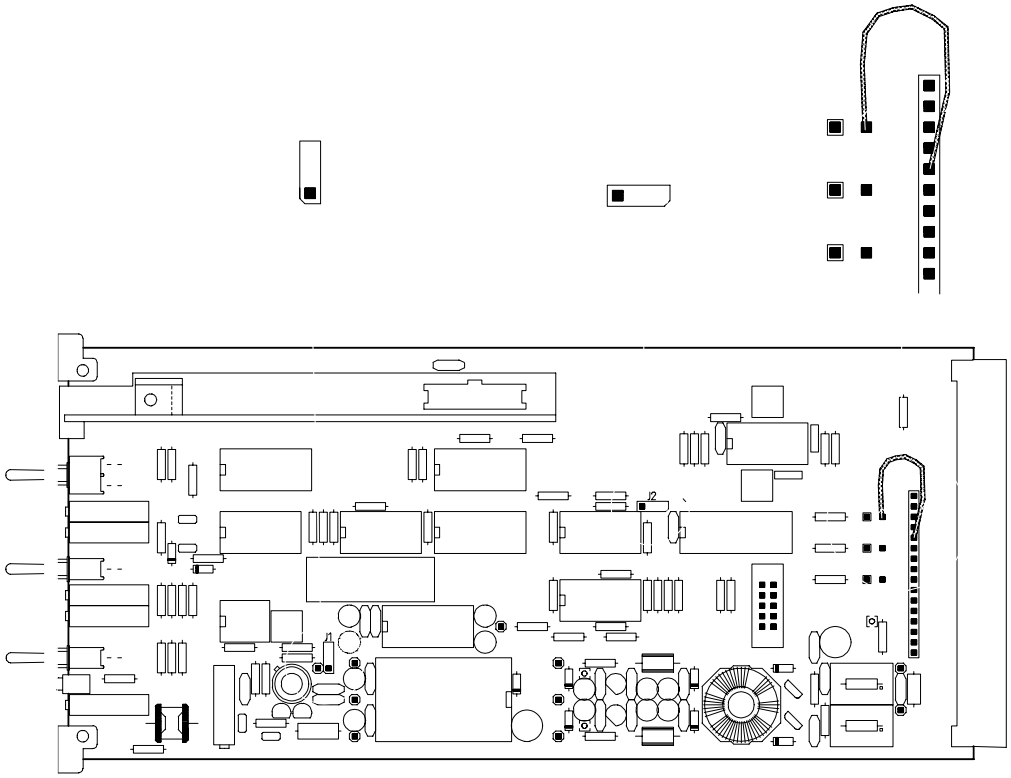


Fig. 2: Position of the internal links

In example C shown above the signal output AV_PH has been set so that the analogue pH signal is on bus line AV 5. The jumper J1 (A) is set so that the input filter is set to 0.2 Hz. The mV signal is not reversed,

Jumper J2 (B).

4.12 Signal output AV_MV to transfer mV signal to PLUGSYS bus system

The link AV_MV transfers the potential measured at the pH electrode to the PLUGSYS bus after multiplication by a factor of 10.

If this voltage is required the link must be set to the required channel (AV1 - AV16). When the module is supplied from the factory this link is in the parking position. The signal is transmitted through the PLUGSYS bus to a recorder or a data acquisition system.

Please note:
This signal is affected by the Jumper J2 **(Fig. 2 B)**. With this jumper the mV signal from the eletrode can be reversed.

Jumper right --> signal not reversed (normal setting on delivery)
Jumper left --> signal reversed

See also Fig 2 B.

pH	Input Voltage [mV]	Output. [V] AV_MV Jumper right	Output AV_MV reversed [V] Jumper left
6	+55	+0.55	-0.55
7	0	0	0
8	-55	-0.55	+0.55

4.13 Signal output AV_MV_SU for transmitting either pH or mV to the bus system

The link AV_MV_SU permits switching the pH or the mV value to one of the 16 possible analogue lines. This link transfers either the pH or the mV signal to the PLUGSYS bus, depending on the position of the **pH/mV** switch. Here again the link must be plugged in the required channel AV1 - AV16. The signal is then passed through the PLUGSYS bus to a recorder or data capture system. When the module is supplied the link is in the parking position.

Please note:
This signal is influenced by the switch **pH/mV**, and in position **pH** also by the **SUPPRESSION** switch.
In position pH the details of Section 4.11 apply.
In position mV the electrode potential is output after multiplication by a factor of 10.

4.2 Filtering the electrode signal, filter setting with Jumper J1 (Fig.: 2A)

Two low-pass filters are available for use with the electrode signal:

Jumper J1 up --> filter 0.2 Hz (time constant 5 seconds)
Jumper J1 down --> filter 0.8 Hz (time constant 1.25 seconds)

As the average reaction time of a mini pH electrode is 10 seconds both ranges can be used. It is recommended to use the 0.2 Hz setting (factory setting) since the indication is more stable here and does not jump continually. See Fig 2 A.

5. Starting up

After the pH electrode has been prepared and the pH electrode cable has been connected to the input socket the housing can be switched on and measurement can begin.

5.1 Calibrating the pH electrode

The description below assumes that a pH electrode Type 830 and a thermostated flow-through chamber (HSE Chemical Electrode Chamber) are being used.

Basic principle of calibrating pH electrodes:
In order to avoid measurement errors it is important that calibration takes place at the same temperature as the subsequent measurements. The entire measurement in the electrode chamber depends on flow rate and it is therefore important that the pumping speed of the roller pump is the same during calibration and during measurement. The recommended flow rate through the chamber should be between 1 and 2 ml/min (check pump output volumetrically). In principle the calibration should be repeated daily.

Procedure:
It is assumed that the circulation thermostat has already been operating for at least 5 min and the electrode chamber has warmed up. In order to ensure chemical stability the electrode should already have been stored in electrolyte solution for at least 24 hours.

(1) Connect the pH electrode to the pH meter, socket **"ELECTRODE FLOATING INPUT"**.

(2) Set switch pH/mV to pH.

(3) Place approx. 3 - 5 ml buffer solution pH 7.00 into a small vessel.

(4) Place approx. 3 - 5 ml buffer solution pH 4.00 into a small vessel.

(5) Hold the input tubing of the electrode chamber into the vessel with pH 7 buffer solution. Where precision measurements are required, the buffer solution should also be warmed to the chamber temperature. It should be noted that the pH buffer solutions are somewhat temperature dependent (see label on the bottle).

(6) Start up the roller pump. Discard any mixture of buffer solution with other solutions in the system. As soon as chamber plus tubing contains only pH 7 buffer solution, circulate the pH buffer solution in a closed loop.

(7) Using a screwdriver set the trimmer "OFFSET at pH7" so that the reading is 7.00. Check the reading over about 30 seconds and adjust it if necessary. The pH should not fluctuate (±0.01 pH).

(8) Remove the inlet tube and hold it in air, so that a airbubble of 3 to 4 cm length in the tube is produced. Stop the roller pump. This airbubble seperates the two calibration solutions to avoid mixtures.

(9) Hold the inlet tubing of the electrode chamber into the vessel with pH 4 buffer. (Note: Calibration could also be performed with pH 10 buffer but the otherwise linear pH graph is slightly deflected above pH 9.5 because of the alkali error).

(10) Start the roller pump. As soon as the system contains only pH 4 buffer solution this is pumped in a closed loop.

(11) After a new pH has become established (approx. 30 sec), adjust the "Slope" trimmer with a screwdriver to set the reading for pH 4 to 4.00.
The pH reading should no longer fluctuate (±0.01 pH).

As a check the two calibration points should be checked again alternatively. In case of larger deviations repeat steps 5 to 11.

5.2 Setting the pH simulation values for calibrating a recorder or computer system

Calibration of a recorder or data acquisition system always requires two calibration points. Since the entire calibration procedure is rather time-consuming a simulation device is incorporated in the pHMM module to simplify the operation. It simulates two pH values and their output voltages.

If e.g. the measurement is to cover the range between pH 6 and pH 8, the simulation values are set to pH 6 and pH 8.

Procedure:

Move the switch **"SIMULATE 1 / MEAS / SIMULATE 2"** to **SIMULATE 1** (right) and hold it in that position. Adjust the reading to 6.00 with the **SIMULATE 1** trimmer using a screwdriver.

Move the switch to **SIMULATE 2** (left) and hold it in that position. Adjust the reading to 8.00 with the **SIMULATE 2** trimmer using a screwdriver.

If now the switch is moved to **SIMULATE 1** (right), the same value as if the pH electrode measures pH 6 is now simulated on the output. In position **SIMULATE 2**, pH 8 is simulated at the output.

The switch automatically returns to the **MEAS** position but the simulated value is retained for a further 10 seconds on the display and at the output.

5.3 Arranging a pH scale on the recorder

After the simulation values have been set as indicated in Section 5.2 the recorder can readily be calibrated.
It is now assumed that the PLUGSYS housing is switched on and ready for use and that a recorder with 8 cm writing width per channel is connected up.
It is of course possible to use some other recorder with a different writing width and chart scaling. It is however necessary to have adequate sensitivity of at least 1 Volt for full-scale deflection.

Assumptions:	
Required pH range:	pH6 bis pH8
Writing width:	80 mm
Chart scale grid:	every cm and mm

With these conditions the simulation values of the pHMM should be set to pH 6 and pH 8. See the example in Section 5.2. The recording span can now be adjusted accurately to fit the chart scaling. A distance of 8 cm is available, i.e. the pH 6 line is positioned at the bottom edge and the pH 8 line at the top edge. The midpoint (i.e. 4 cm) corresponds to pH 7, so that 1 cm represents 0.25 pH.

Follow the procedure indicated below:

(A) Position the recorder pen at the bottom edge of the chart. Zero the recorder input. Set the pen to the zero line.

(B) On the pHMM set the switch **pH/mV** to **pH**. Set the **SUPPRESSION** switch to **ON**. The green LED above ADJ is alight.

(C) Start the chart drive. Move switch **SIMULATE 1 / MEAS / SIMULATE 2** to **SIMULATE 1** and hold it there. The display shows 6.00; pH 6.00 is being simulated.
After releasing the switch the value is maintained for a further 10 sec. Since the adjustment probably takes somewhat longer it is best to hold the switch in that position. Now adjust the pen to zero with trimmer ADJ using a screwdriver. The zero line now corresponds to pH 6.

(D) Move switch **SIMULATE 1 / MEAS / SIMULATE 2** to **SIMULATE 2** and hold it there. The display shows 8.00; pH 8.00 is being simulated.
Now adjust the recorder gain so that the pen is at the top edge of the chart (8 cm). This value corresponds to pH 8.00.

(E) Checking the adjustment:
Run the recorder at a slow speed. Switch **SIMULATE 1 / MEAS / SIMULATE 2** on **MEAS**. The value measured by the pH electrode is being indicated and shown on the chart. Move the switch briefly to **SIMULATE 1**. The value pH 6 is now being simulated for 10 sec, the pen is against the bottom edge of the chart. Next move the switch to **SIMULATE 2**. pH 8 is now being simulated for 10 sec, the pen is at the top edge of the chart.

By quickly keying in the simulation values it is possible at any time (also during an experiment) to check the recorder settings.

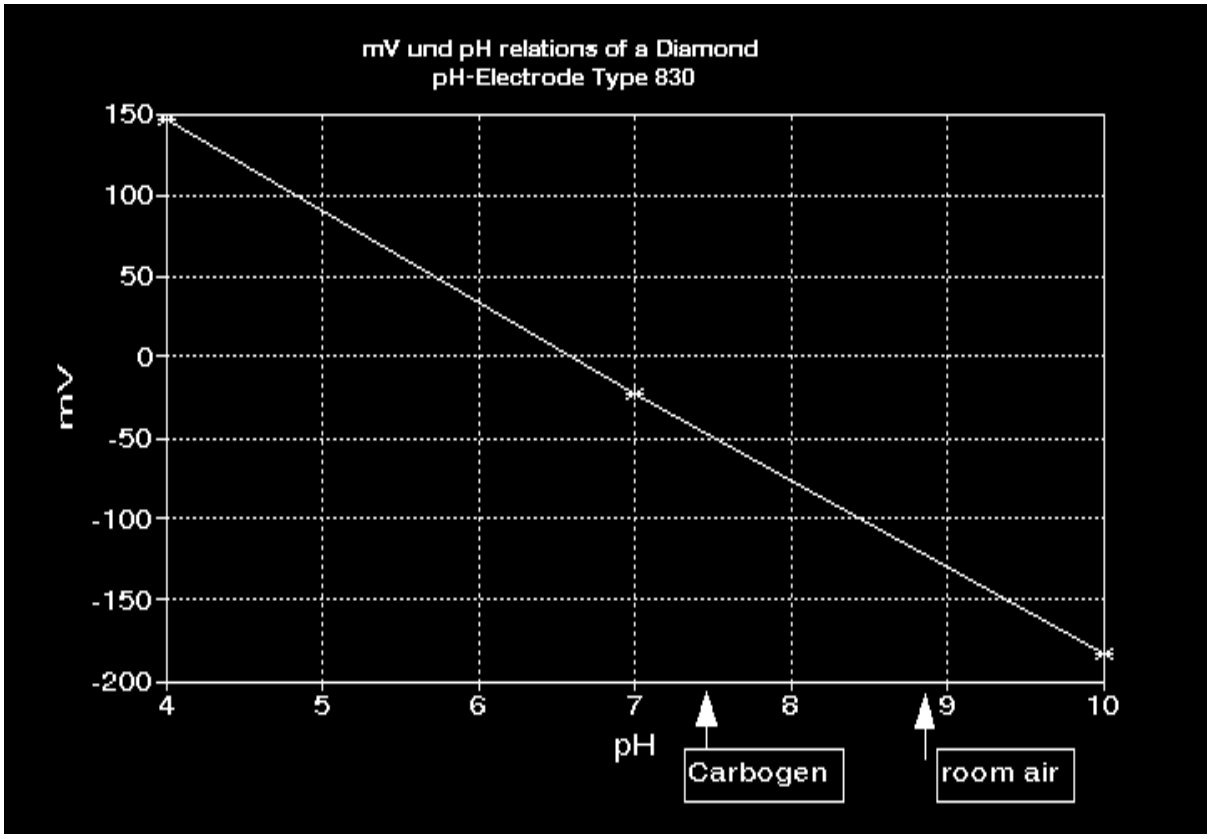
Please note:
Simulation does not replace the calibration! Simulation only provides output values for a recorder. During a measurement these are only correct if the electrode has been calibrated correctly!
A pH electrode can only be calibrated by the use of buffer solutions! See Section 5.1.

Calibration or checking of calibrated values should be performed at least once a day. The better method is to follow GLP and check before and after each experiment.

5.4 Experiment

If the pH electrode has been calibrated the measurement can now be started under the same conditions (constant temperature, constant flow).
The reaction time of the electrodes is about 10 seconds (combination pH electrode Type 830). The electrode has a stability of 0.05 pH/day. The electrode slope is approx. 55 mv/pH.
Please note: During the experiment it is important to ensure that there are no air bubbles in the electrode chamber.

5.5 pH-mV-Relations of the pH-Elektrode Type 830



The diagram above shows the pH-mV-relation of a combination pH-electrode Type 830. An increasing pH value results in a decreasing electrode voltage. This electrode had a offset voltage at pH7 of -22mV. A optimal pH electrode shows at pH7 a value of 0mV. Because of tolerances during manufacturing are values of ±20mV normal. This offset is set to zero in the calibration procedure with pH7 buffer with trimmer "OFFSET at pH7". The kurve is shifted parallel. The Slope of the elecrode is about 57mV/pH (172mV/3pH). The mean value of the electrodes type 830 is at 55mV/pH. In the calibration procedure first pH7 is set, this means that the kurve is electronically
adjusted to zero. After that "Slope" has to be adjusted.

6. Input

The **pHMM** module carries a BNC input socket for combination electrodes. Alternatively, electrodes with an external reference electrode can be used; the reference electrode is connected to the black input socket EXTERNAL REFERENCE (2 mm socket).
The input is floating, i.e. isolated from ground. This is necessary in order to prevent mutual interference if

several electrochemical values such as pO_2, pCO_2, Na^+, K^+ oder Ca^{++} are being measured continuously at the same time.
Isolation also prevents hum interference. The input circuit is isolated from the output circuit and the housing by an isolating amplifier.

7. Description of the controls

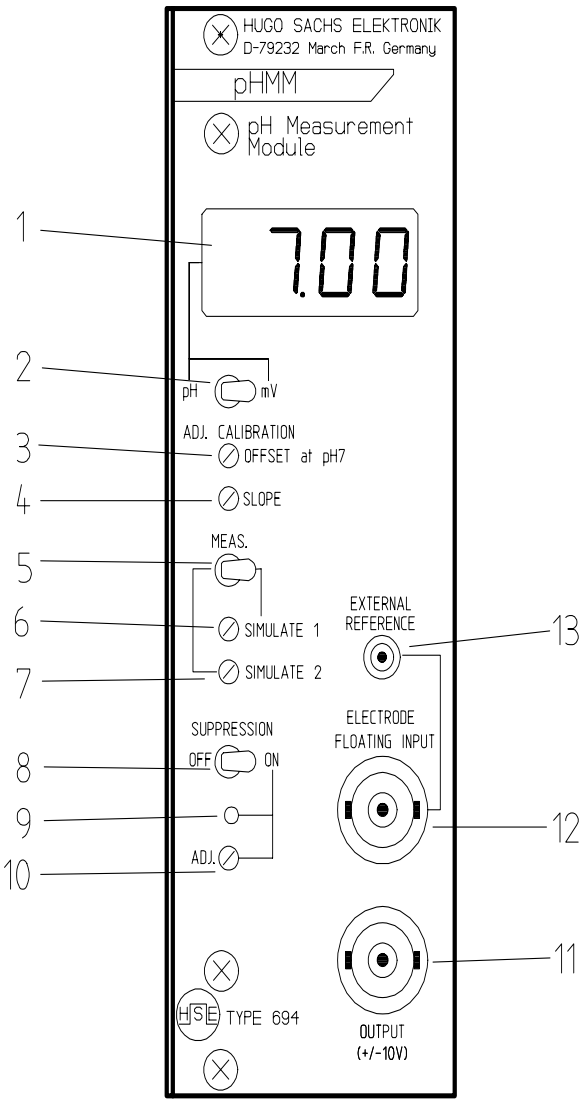


Fig.3 Controls on the front panel

(1) Display to indicate pH or mV value. The display is switched over using the **pH/mV** switch.

(2) **pH/mV** switch. This switch selects either **pH** or **mV** indication on the display. In position **pH** the measured pH is indicated with two decimal places. In setting **mV** the measured mV value is indicated. The BNC socket OUTPUT carries, depending on the switch setting, either a voltage corresponding to the pH, or a voltage corresponding to the measured mV (mV x 10).

(3) Trimmer **ADJ.CALIBRATION, OFFSET at pH7**. This trimmer is used to adjust pH 7.00 during calibration of the pH electrode. During this operation the electrode must be immersed in a pH 7 buffer. See also Section 5.1, Calibration.

(4) Trimmer **ADJ.CALIBRATION, SLOPE**. This trimmer is used to adjust pH 4.00 during calibration of the pH electrode. During this adjustment the electrode must be immersed in a pH 4 buffer. See also Section 5.1, Calibration.

(5) Switch **SIMULATE 1 / MEAS / SIMULATE 2**. This switch automatically returns to the central **MEAS** position and is used to simulate two freely selected pH values in order to simplify the calibration of a recorder or data acquisition system. If the switch is pushed to the right to **SIMULATE 1**, the pH value set on the trimmer **SIMULATE 1 (6)** is shown on the display and appears at the output. After releasing the switch it jumps back to its central position; the simulated pH value is however held for a further 10 sec approx. in order to permit readjustment of the recorder without having to hold down the switch continuously. In the left position **SIMULATE 2** the pH value set on the trimmer **SIMULATE 2 (7)** is shown on the display and appears at the

output. Here again the simulated pH is held for a further 10 sec approx.

(6) Trimmer **SIMULATE 1**: This trimmer is used to set the first pH simulation value. To do this, push the switch **SIMULATE 1 / MEAS / SIMULATE 2** to the **right**, hold it there and, using a screwdriver, set the required value as shown on the display. See also Section 5.2.

(7) Trimmer **SIMULATE 2**: This trimmer is used to set the second pH simulation value. To do this, push the switch **SIMULATE 1 / MEAS / SIMULATE 2** to the **left**, hold it there and, using a screwdriver, set the required value as shown on the display. See also Section 5.2.

(8) Switch **SUPPRESSION OFF/ON**. Using this switch the range suppression can be switched on and off. This function is required when using a recorder or data acquisition program.
If the **SUPPRESSION is OFF**, a signal of 1 Volt per pH appears at the internal output AV_PH and at the BNC socket on the front panel; at pH 7 the output is 7 Volt, at pH 4 correspondingly 4 Volt. The physiological range is about pH 7.4. To show for example the range from pH 7 to pH 8 on the recorder leads to difficulties since the position control for the recorder pen can not be adjusted far enough so that pH 7 can be shown at chart zero. This problem is avoided by switching on the SUPPRESSION function. If the **SUPPRESSION** switch is set to **ON** the output voltage range can be shifted with the **ADJ trimmer**; pH 7 can then be placed on 0 Volt. In this way the range from pH 7 to pH 8 can be shown enlarged on the recorder. See also Section 5.3.

(9) LED **"SUPPRESSION ON"**. This LED lights up as soon as SUPPRESSION is switched on.

(10) Trimmer **ADJ**. This trimmer is used to adjust the range suppression. To set pH 7 to recorder zero, for example, the SUPPRESSION must be switched on. Then pH 7 can be simulated with SIMULATE 1. Using the ADJ trimmer the recorder pen is then set to the required position.

(11) BNC socket **OUTPUT**. This output socket carries, depending on the setting of switch pH/mV, either the pH or the measured mV (electrode voltage x 10).
If SUPPRESSION is switched on, this socket carries the pH signal with range suppression.

(12) BNC socket **ELETRODE FLOATING INPUT**. This socket is used to connect the electrode.

(13) 2 mm socket **EXTERNAL REFERENCE**. This socket is used to connect an external reference electrode when using a pH electrode with separate reference electrode.

8. Faults, causes and remedies

Fault:
pH fluctuates strongly or appears unrealistic.
Cause:
Air bubble at the electrode tip, electrode chamber not grounded.
Remedy:
Produce a large air bubble, e.g. by briefly opening the supply tubing. The large air bubble produced (approx. 20 - 30 mm long in the supply tubing) then joins up with the undesirable bubbles in the chamber and can be pushed out by the liquid following behind. Connect the chamber to the central ground point.

Fault:

Electrode slope appears very steep (normally approx. 55 mV/pH).
Cause:
Electrolyte exhausted; possibly due to large negative pressure by closing or kinking the tubing before the electrode chamber, or measurement in highly acidic or highly alkaline media.
Remedy:
Refill the electrolyte using the syringe supplied.

Fault:
Measurement completely incorrect, instrument overloaded.
Cause:
Ground not connected.
Remedy:
Connect ground cables to central ground point.

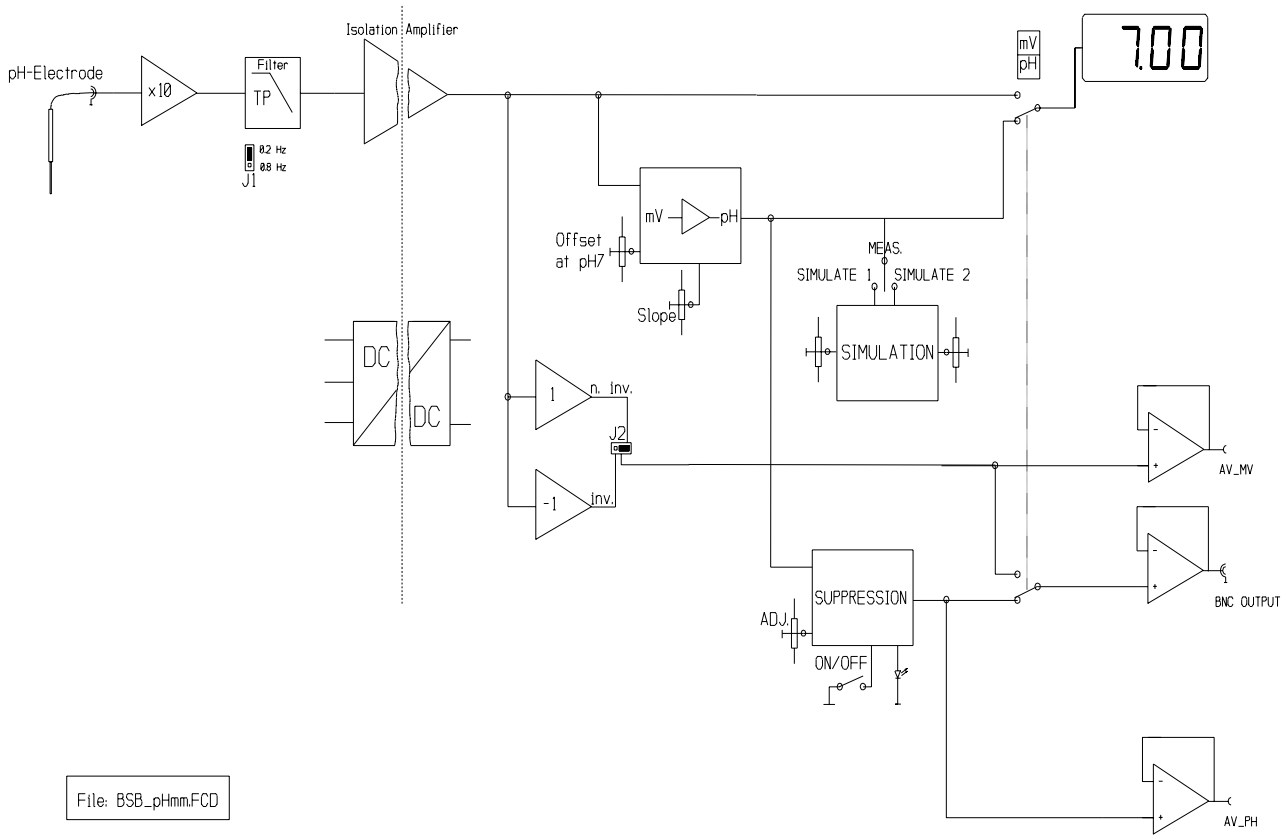
9. Maintenance and cleaning

The PLUGSYS module does not really require any maintenance. The **pHMM** module is supplied fully calibrated, only the pH electrode still requires calibration. Any operation on or modification of the electronic circuit invalidates the warranty and the manufacturer's product liability.
The front panel can be cleaned if necessary with a lightly moistened (not a wet) cloth. Before cleaning always pull out the mains supply plug!
No moisture must find its way into the unit and especially not into the switches and keys, since this leads to corrosion at the switch contacts resulting in faulty operation. In general the PLUGSYS housing should be protected against splash water and salt solutions as this may damage individual components and may cause a short-circuit!

10. Transport and storage

In order to avoid transport damage when returning the unit to the factory, the PLUGSYS housing should be packed in a suitably large carton (the carton should allow a spacing of about 10 cm all round so that sufficient packing material such as polystyrene, hard foam panel or similar can be included to protect against impact damage). When shipping individual modules these should also be well packed, preferable enclosed in antistatic foil or envelope.

11. Block diagram of the pHMM module



12. Technical data

Input:	isolated differential input, max isolation voltage 500 V
Input socket:	BNC
Input impedance:	10^{15} Ohm
Input current	±300 fA
pH range:	0 to 14 pH
Resolution:	0.01 pH
Indication:	3 1/2 digit LED display
Millivolt range:	±600 mV
Offset range at pH 7:	±100 mV
Slope:	45 mV/pH to 90 mV/pH
Output:	1 Volt per pH at BNC socket OUTPUT on front panel (±10 V 5 mA max.) The output voltage for pH and mV is also available internally on the PLUGSYS bus.
Suppression:	OFF --> output voltage 0 Volt at pH 0 and 10 Volt at pH 10 ON -> output voltage 0 Volt can be adjusted within the pH range 0 to 14
Calibration:	2-point calibration with standard buffer solutions
Simulation:	two physiological measurement points for calibrating a recorder can be set within the range 0 - 14. A switch is used to switch the simulation values to the display and the output.
Recorder outputs:	the internal output AV_PH is connected through a link to the PLUGSYS bus system. The pH signal is connected to a recorder through the Recorder Output Module installed in the PLUGSYS system. Direct connection at the BNC socket on the front panel is also possible.
Ambient conditions:	Operating temperature: 10 to 40°C
Relative humidity:	20 to 80% without condensation
Storage temperature:	-20 to +60°C
Supply:	5 V 450 mA, supply via the PLUGSYS system bus
Mechanical data:	
Dimensions:	module for PLUGSYS housing width 8 E (40.8 mm) height 3 U (128.7 mm) depth Eurocard (220 mm)
Connectors:	DIN 41612, 64-pin VG connector BNC
Weight:	400 g
Accessories:	BNC output cable and Operating Instructions

Appendix

Standard buffer solutions are required for calibrating the pH electrodes. The solutions are available in the following pH values:

2.00
4.01
7.00
9.21
10.00

Buffer solutions to NBS and DIN 19266 are available in the following pH values:

4.008
6.865
9.180

Standard buffer solutions as indicated above are available from the following suppliers (in Germany):

Ingold Messtechnik GmbH
(Mettler-Toledo Prozessanalytik GmbH)
Siemensstrasse 9
D-61449 Steinbach/Ts, Germany
Phone (international): +49 6171 700 10
Fax (international): +49 6171 700 199

Radiometer Deutschland GmbH
P.O.Box 3
D-47862 Willich, Germany
Phone (international): +49 2154 818 0
Fax (international): +49 2154 818 184

Local representatives of Ingold (Mettler) and Radiometer should also be able to supply these buffer solutions.