

pH-detector PHI 04D

USER'S MANUAL



0 Introductory:

pH-detector pHI 04D is a digital instrument developed for accurate potentiometric monitoring, especially for measuring pH by a glass electrode and ionic concentrations by means of ion-selective electrodes. The instrument enables the measured values to be controlled by analog signal output or to be stored in memory and sent to a computer for subsequent processing or printing. The measured value may be sampled at preset time intervals and its time dependence is shown on the instrument display.

0.1 Operating Modes of pHI 04D:

- 1) Measurement of pH with analog output to 0.003 pH units
- 2) Measurement of pH read to 0.001 pH units
- 3) Measurement of voltage within the range ± 1200 mV read to 0.1 mV (option)
- 4) Measurement of ion concentrations by ion-selective electrodes (option)
- 5) Measurement of temperature (option)

The measured values can be stored in memory and subsequently displayed anytime. The set of measured values can be printed or sent to a computer for potential further processing.

When used to measure pH PHI 04 D can be calibrated either by standards of known pH, to be entered on the instrument memory, or by standards with accurate pH values of 4.01, 7.00 and 10.00; in the latter instance the instrument is able to determine from the input voltage which calibration standard is currently measured and assigns the appropriate value to the input voltage.

The calibration data are stored in memory. When ionic concentration is measured by means of ion-selective electrodes after previous calibration, the instrument displays the determined concentration in the selected units. The instrument may be calibrated simultaneously for a glass electrode and for 6 different ion-selective electrodes. The graphic display enables the time dependence of the currently measured quantity to be shown.

0.2 Fundamental Technical Parameters

Power:	Power adapter 12 V
Power input:	3.6 VA
Internal resistance:	10^{12} Ω minimum
Measured values:	pH voltage (with a fixed or shifted scale) concentration - 6 ranges temperature (platinum thermometer, option)
Precision of pH measurement:	read to 0.001 pH units, error in pH 0.002 units
Precision of voltage measurement:	read to 0.1 mV, precision 0.2 mV
Precision of concentration measurement:	0.25 % of the measured value
Analog output	0 – 1 V DC
Precision of temperature measurement:	read to 0.1 °C
Additional functions:	storing measured values in memory, sampling values at pre-set time intervals displaying graphically the time dependence of measured values,printing results, sending results to a computer

1 Instrument Description

1.1 Control Elements of the pH- detector PHI 04D

Figure 1 shows the front panel of the instrument. The alphanumeric display (1) indicating the results of measurement is situated in the upper part of the front panel. The connectors for individual sensors are in the lower part. The pH or the ion-selective cells are connected through the BNC connector marked 'pH' (2). In the event a combined electrode is not used the reference electrode is connected by means of connector (3) marked 'Ref'. Connector marked 'PT100' is used to connect a platinum resistance thermometer. If connected, the instrument displays the temperature and performs automatic temperature compensation; otherwise the temperature of the measured solution must be entered manually.

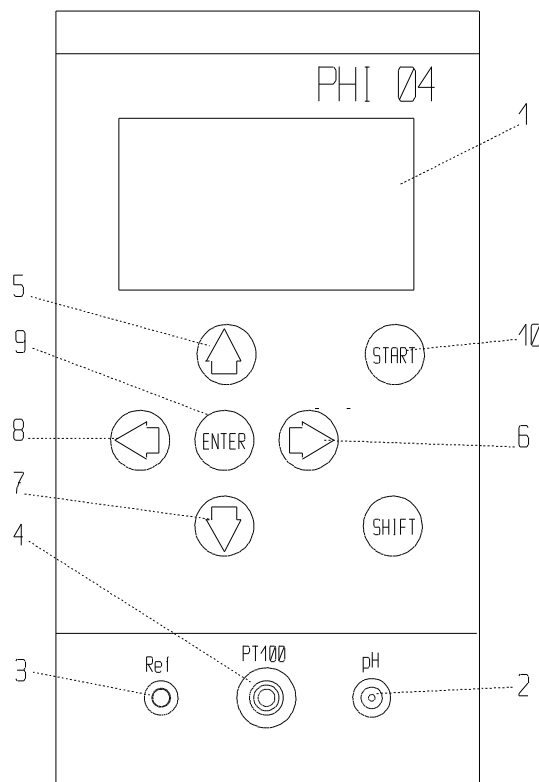


Fig. 1 Front panel of PHI 04D

The buttons situated below the display are used to control the instrument and enter numerical values in memory. The ENTER button (9) is used in instrument calibration to accept the selected value of a numerical parameter and store it in memory. In the base operating regime of the ionometer, where the measuring method is selected, as well as in higher operating regimes, where additional modes of operation are selected from the menu, this button confirms the selection and at the same time selects the next operating regime.

The arrow buttons (5), (6), (7), and (8) have several functions:

- 1) In instrument setup and calibration they are used to select the value of a numerical parameter to be entered. The numerical value to be stored in the microprocessor memory by pressing the ENTER button (9) is then shown in the bottom-right part of the display and the cursor is situated at one of the digits. That digit can be then varied by buttons (5) and (7): press (5) to increase the digit by one or (7) to decrease it. It is important to note that, since these buttons add or subtract one from the given order of magnitude, in instances

where you press button (7) to change digit '0' or button (5) to change digit '9' the change is reflected in the next higher order of magnitude.

Buttons (6) and (8) shift the cursor in the corresponding direction, thus enabling one to set all valid digits of the number to be entered.

2) Selection from the list of operating regimes is made by means of buttons (5) and (7). All items are then arranged circularly and can be browsed by buttons (5) and (7) in the corresponding direction. In the base operating regime selected after the ionometer is switched on, the buttons are used to select the operating mode, e.g., measurement of potential, pH, or concentration by means of an ion-selective electrode, etc. and, once the operating mode is selected, they are used to select one of the submenu items. In the operating mode "Cancel calibration" they select one of two alternatives: No – retain the calibration, Yes – cancel the calibration. Since there are only two possibilities, both buttons are here equivalent and, when any of them is pressed, the choice indicated on the display is rejected and replaced by the alternative.

3) Searching the memory for the stored value of a given serial number.

4) Button (8) transfers the instrument from a higher operating mode to the base mode.

The PRINT button (10) directs the instrument to print the actual value of the measured quantity. The SHIFT button (16) on the front panel enters the measured value in memory, provided the regime of manual storing data is set; in the regime of sampling at regular time intervals, pressing the button initiates the operation.

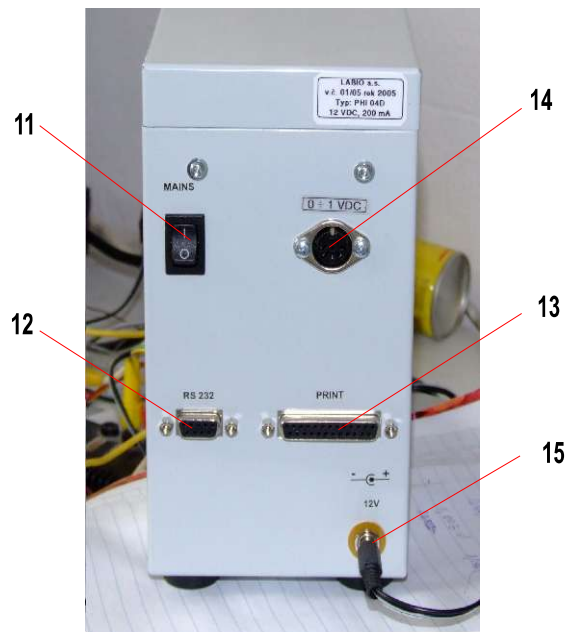


Fig. 2 Rear panel of pH-meter and ionometer PHI 04D

The power switch (11), the R232 connector for connecting the ionometer to a PC (12), the connector for a printer (13), the analog output 0 – 1V DC(14), and a cable leading to a power adapter (15) are situated on the rear panel (Fig. 2).

1.2 Instrument Display

All measured values are shown on the display depicted in Fig. 3.

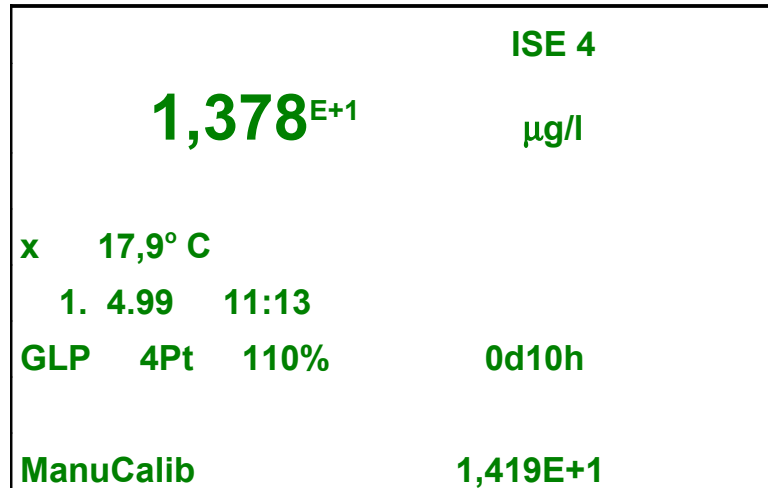


Fig. 3: Display of PHI 04D

The measured value is highlighted in the upper part of the display - as a decimal number when voltage or pH is measured or in the semi-logarithmic format in measurement of ion concentration.

The type of measurement currently performed is indicated in the upper right corner : mV, pH, ISE1 ... ISE6; when ion concentration is measured, also the units in which the ion concentration displayed is expressed are shown under the mark of measurement type.

In all operating regimes the measurement temperature is shown on the first left line under measured value: if the thermometer sensor is connected *via* connector PT100, the displayed value indicates the temperature actually measured, otherwise it is the last entered value of temperature at which the temperature compensation is to be performed; the latter instance is indicated by letter 'x' shown to the left of the displayed temperature. In line 2, below the temperature, the display indicates the date and time. When pH or ion concentration is measured by an ion-selective electrode, line 3 shows important information indicating whether the data from last calibration are still applicable. If this is still possible according to the ISO standard, 'GLP' is displayed on the left-hand side of line 3, and information about the number of standards used in the calibration is shown to the right, followed by information about the slope of the dependence of the electrode voltage on pH or pX, expressed in per cent of the theoretical slope. Time from the last calibration is indicated at the right in line 3.

Indication of the operating regime of the instrument, if different from the base regime, is displayed in the last line, at the bottom left corner. If there is no value displayed in the bottom right corner, one may select the operating menu by means of buttons (5) and (7). If a numerical value to be entered in memory is being selected, it is shown to the right in the last line and, after it is entered in memory by pressing ENTER, it is replaced during calibration by the number of data points currently comprising the calibration dependence.

The above description refers to the display in the operating regime where the measured value is shown. If the instrument is switched to the mode where a time dependence of the measured value is to be displayed (by selecting the menu item Log and confirming item Graph), the display shows the x and y co-ordinates and below, in the event values of pH or voltage are sampled, also indication of the range set for each of the two co-ordinates. The value at the abscissa (x) indicates the serial number of the data point (the full range

corresponds to 127 data points) as shown to the left; the pre-set range referring to the ordinate (y) is displayed on the right-hand side – the range is automatically adapted during the measurement. By pressing ENTER again the information concerning the range below the abscissa is replaced by information about the currently selected menu item.

The pH-meter may be calibrated also in the graphical mode but this procedure is not recommended. To leave the graphical mode select the Log menu item by means of the arrow buttons (5) and (7) and press ENTER to display 'Graph'. Upon pressing ENTER once more the graphical mode is abandoned and the instrument switched to the normal operating mode where the display shows the measured value.

2. Measurement with the pH-detector PHI 04D

2.1 Preparation for pH measurement with automatic temperature compensation:

1) If the pH meter is in the graphical mode, switch to the alphanumeric display mode as described in section 2.1.1. Using buttons (8) and ENTER (9) switch the ionometer pH 04 to the base operating regime, where the bottom line is empty, and then to the "pH measurement" mode by means of buttons (5) and (7).

2) Connect the platinum thermometer sensor *via* connector PT1000 (4). Connect a glass electrode to connector pH. Unless a combined pH electrode is used, connect the reference electrode *via* connector Ref (3).

3) Rinse the reference and the pH electrode with distilled water and dry.

Note: To preserve its characteristics the glass electrode must not be allowed to dry; if not in use for some time it should be kept in distilled water containing a small amount of a pH 7.0 buffer solution. The sintered disc of the reference electrode that ensures a conductive connection with the electrolyte solution should not be allowed to dry. A freshly wetted reference electrode may change its potential; it is therefore advisable to keep it in the saturated solution of the internal electrolyte.

2.2 Calibration for pH measurement with automatic temperature compensation:

Prepare the instrument for pH measurement as described in section 2.2.1 and then proceed as follows:

1) Press ENTER (9) and by means of the (5) and (7) buttons select the ClearCalib item from the submenus shown in the last line.

2) Press ENTER (9). 'No' appears alongside ClearCalib in the last line. Press (5) or (7) (it is immaterial which in this case). 'No' is replaced by 'Yes'. Press ENTER (9) to confirm cancellation of the previous calibration. The numerical value displayed in line 1 representing the pH value disappears and is replaced by

-.--- pH

displayed to the right of 'pH'.

3) Press ENTER (9) and, using buttons (5) and (7), select item ManuCalib from the submenus shown in the left bottom corner.

4) Rinse the measuring electrodes with distilled water, dry and immerse in a buffer solution of known pH.

5) Press ENTER (9). A number is displayed in the bottom left corner. Using buttons ⇒ (6) and ⇐ (8) to shift the cursor and buttons (5) and (7) respectively to increase and decrease by one the displayed digit, set the number to the pH value of the standard in which the

measuring electrodes are immersed, and confirm by pressing ENTER (9). 'Point 1' is displayed at the bottom right corner to indicate that the first point of the calibration set has been just entered.

6) Press (8) to return to the base state of the ManuCalib submenu. Prepare the next buffer solution, rinse the electrodes with distilled water and dry, immerse them in the buffer solution, enter its pH value in the same manner as before and confirm by pressing ENTER (9). Once the display confirms in the bottom right corner that calibration has been effected with the next standard, switch the pH meter to the base state. If multiple-point calibration is desired, proceed in the same manner with additional standards.

Note 1: If a multiple-point calibration is used, the pH ionometer calculates the pH value from voltages corresponding to pH values of buffer solutions bracketing the current input voltage.

Note 2: Calibration for measurement of both pH and ionic concentration by means of the ion-selective electrode can be performed by no more than five standards. An attempt to store in memory a sixth value of pH corresponding to the input voltage results in the message 'MemFull' being displayed to the left of 'ManuCalib'.

2.3 Autocalibration in pH measurement:

Calibration of the pH ionometer can be expedited if buffer solutions with accurate pH values of 4.01, 7.00, and 10.00 are at disposal. Their pH values need not be entered since pHI 04 can identify them on its own.

In autocalibration of the pH meter proceed as follows:

- 1) Prepare the pH-meter for measurement as described in section 2.2.1.
- 2) Using the procedure described in items 1 and 2 of section 2.2.2 delete the preceding calibration: '---.--- pH' must be displayed in the first line.
- 3) Press ENTER and by means of buttons (5) and (7) switch the instrument to the submenu item 'AutoCalib', displayed in the bottom left corner.
- 4) Rinse the electrodes with distilled water, dry with a cellulose wad and immerse in the solution of the 1st calibration standard (any of the buffer solutions with pH 4.01, 7.00 and 10.00 at 25 °C). Press ENTER (9). If the input voltage corresponds to pH 4.01, 7.00 or 10.00, 'Point 1' appears in the bottom right corner of the display; otherwise the input voltage does not correspond to any of the three standards and autocalibration is not possible.
- 5) Press (8). Prepare the next buffer solution and enter in memory the input voltage corresponding to its pH in the same manner as before.

2.4 Setting the isopoint pH:

- 1) Switch the ionometer to the base operating regime by means of buttons (8) and ENTER (9).
- 2) Using buttons (5) and (7) switch the instrument to the "pH measurement" mode.
- 3) Press ENTER (9).
- 4) By means of buttons (5) and (7) identify submenu Set Iso-pH and confirm by pressing ENTER (9). By means of buttons (5), (6), (7) and (8) set the number with the cursor that appeared in the bottom right corner to coincide with the pH value of the iso-point and confirm by pressing ENTER (9). By pressing (8) switch the instrument to the base operating mode of the function "pH measurement".

2.5 Setting the temperature in manual temperature compensation:

- 1) Disconnect the platinum thermometer sensor.
- 2) By means of buttons (8) and ENTER (9) switch the ionometer to the base operating regime.
- 3) Use buttons (5) and (7) to switch the instrument to the function "pH measurement".
- 4) Press Enter (9).
- 5) By means of buttons (5) and (7) identify the SetTemper submenu item and press ENTER (9). Cursor appears in the number indicating the temperature (18); the number can be now changed by means of buttons (5), (6), (7) and (8) – set the actual temperature of the solution and accept by pressing ENTER (9). That temperature will be used to calculate pH from the input voltage.
- 6) Press (8) to switch the instrument to the base mode of the "pH measurement" function.

2.6 Measuring pH with calibrated detector pHI 04 D:

- 1) Using buttons (8) and ENTER switch the instrument to the base operating regime where the bottom line is empty.
- 2) Using buttons (5) and (7) switch the instrument to the "pH measurement" mode.
- 3) Connect the platinum thermometer sensor or, if need be, enter the solution temperature as described in section 2.2.5.
- 4) Connect the glass electrode and the reference electrode *via* connectors pH and Ref, respectively, rinse the electrodes with distilled water and immerse in the measured solution. After a transient time the display shows the actual pH value.

2.7 Flow through measuring cell

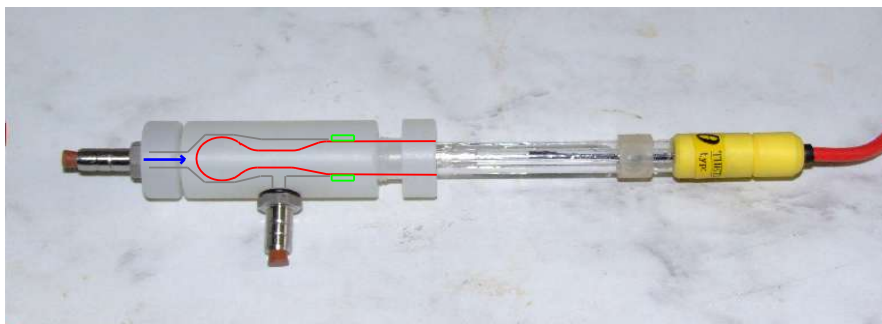


Fig. 4: pH cell view

Flow through measuring cell consists of polypropylene body with stainless steel input and output endfittings. Combined glass electrode is fixed inside by a silicone sealing. Cell volume is less than 1 ml.

Cell is to be used in vertical position with electrode output in most upper position. Electrode is equipped with BNC connector which has to be fixed to detector input on its front panel. Calibration of the instrument is done when proper buffer is inserted into the cell.

2.8 Storing the Measured Value in pH-meter Memory

2.8.1 Entering a measured value in memory manually:

- 1) Set the ionometer pHl 04 MG to measure the desired quantity.
- 2) By means of buttons (5) and (7) select item Log from the base menu and press ENTER. Using buttons (5) and (7) select Setup from the submenu - 'Setup' replaces 'Graph' on the display. Press ENTER, identify the submenu item Manual by means of buttons (5) and (7), and accept by pressing ENTER. The (8) button switches the instrument back to the base operating regime.
- 3) If the value to be stored in memory is displayed, press SHIFT situated in the bottom right corner of the front panel. The right part of the line displaying the temperature, which has so far showed 'Log 0' indicating the lowest free memory register, now displays 'Log 1', since register 0 contains the measured value and register 1 is the next register free, ready to accept the next value entered by the SHIFT button.

2.8.2 Entering a measured value in memory automatically

- 1) Set the ionometer pHl 04 D to measure the desired quantity.
- 2) By means of buttons (5) and (7) select item Log from the base menu and press ENTER. Using buttons (5) and (7) select Setup from the submenu - 'Setup' replaces 'Graph' on the display.
- 3) Press Enter and by means of buttons (5) and (7) select item 00:02 from among the additional submenus. Upon pressing ENTER the instrument will subsequently store the measured values in memory each 2 seconds. The sampling interval may be set by buttons ↑ (5), ⇒ (6), ↓ (7) and ⇐ (8) by shifting the cursor inside the displayed number and modifying individual digits similarly as when entering numerical values during calibration. By pressing ENTER accept the sampling interval set in this manner and return the instrument to the base operating regime by pressing ENTER. Press SHIFT – sampling will thereafter proceed automatically and, once a value is stored in memory, the number of the next free register will be shown to the left of the displayed temperature; in contrast to the regime where data are stored manually, the character 'R' is now shown alongside the register number.

2.8.3 Displaying a graph of measured values

- 1) Switch the instrument to the base operating regime.
- 2) Using buttons (5) and (7) identify item Log displayed in the bottom left corner. Press ENTER twice – co-ordinates of a graph will appear. Values stored in memory, if any, are displayed as individual points of the graph.

When values of pH are measured the bottom part of the graph indicates the range of both the abscissa and the ordinate. During sampling the ranges are adjusted automatically to ensure that all measured values lie inside the quadrant actually displayed.

Note: To switch the display from the graphical to the alphanumeric mode select item Log and press ENTER twice.

2.8.4 Recalling from memory and displaying measured values

- 1) Switch to the alphanumeric display mode by selecting the Log item and pressing ENTER twice. Switch the instrument to the base operating regime by pressing (8).
- 2) Using buttons (5) and (7) select from the menu item Log in the bottom left corner of the display and press ENTER. In the submenu to the right of the display select item Browse by

the same two buttons - 'Browse' will be displayed instead of 'Graph'. Press ENTER. A message characterising the number stored in register 0 is displayed in the bottom line as follows: 'Log' to the right followed by the number of the register currently displayed, the actual value stored in that register and to the right an indication what type of value is involved, e.g. 'Log 0 7.000 pH' indicates that register 0 stores a measured pH value equal to 7.000. The number of the register of interest can be now selected using buttons (5) and (7) and the stored value identified. To cancel the function press (8) – the message disappears and the instrument is switched back to the base operating regime.

2.8.5 Printing measured values stored in memory:

1) Switch to the alphanumeric display mode by selecting the Log item and pressing ENTER twice. Switch the instrument to the base operating regime by pressing (8).

2) Using buttons (5) and (7) select item Log from the menu in the bottom left corner of the display and press ENTER. Select item Print by the same two buttons in the submenu to the right of the display and accept by pressing ENTER. If a printer is connected to the pH-meter *via* connector PRINT situated on the rear panel, one can now print the values stored in memory by pressing START.

2.8.6 Storing the measured values in computer memory:

To process the data on a PC the pH-meter and the computer must be interconnected by the serial port *via* connector R232 situated on the rear panel. A special program is necessary for processing the data on a personal computer.

2.8.7 Erasing the memory registers of the pH-meter and ionometer pHI 04:

To erase data from the memory registers switch the instrument from the base operating regime to the Log menu and accept by pressing ENTER; then identify and select submenu CLEAR and accept the selection by pressing ENTER – all data stored in memory registers will be erased and a message informing that register 0 is now free will be displayed.

Manufacturing, distribution and service

Labio a.s, Heyrovského náměstí 2, 16201 Praha 6, Czech Republic

tel/fax 00420 235 360 074, tel 00420 235 358 044

e-mail: sales@labio.com