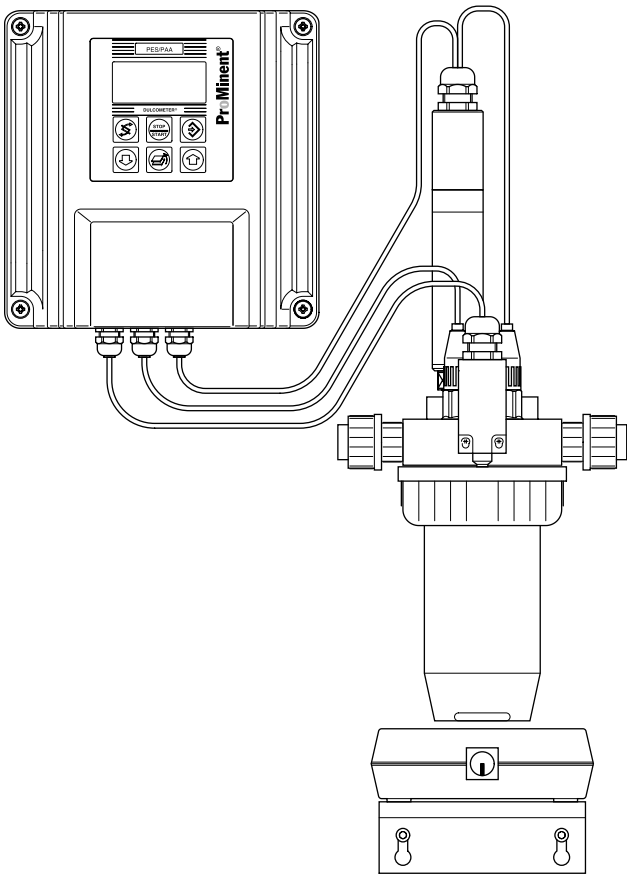
Operating Instructions

DULCOMETER® D1C

Part 3: Adjustment and Operation, Measured Variable H_2O_2 and peracetic acid (PAA)

D1C Schr H202



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Please first read the operating instructions completely! • Do not dicard!
Warranty becomes void in the case of damage caused by operating errors!

Part No. 987936 **ProMinent Dosiertechnik GmbH · D-69123 Heidelberg · Germany** BA DM 061 8/99 GB

1 Contents / General User Information

General instructions for the user

These operating instructions describe the technical data and functions of the DULCOTEST® PEROX sensor signal transmitter PEROX-micro-...-mA and the accessory devices. These descriptions contain detailed safety instructions and are sectioned clearly according to the sequence of action steps. The activities which must be carried out are designated by bold dots (•) .

IMPORTANT:

The instrument must always be in an electrically dead state (disconnected from the mains voltage) while connecting measuring probes and/or accessory devices.

IMPORTANT:

Please note which parts of this manual are relevant for your particular version of the instrument. You can determine this from the equipment designation and identification code.

IMPORTANT:

Correct measuring and metering is possible only when the measuring probes are functioning correctly. The probe must therefore be checked and recalibrated regularly.

2 Measuring principle

Measuring principle

The PEROX measuring system is based on amperometric measurements with some special features not found in conventional systems. The small surface area measuring electrode is made of platinum (for hydrogen peroxide measurements) or gold (for peracetic acid measurements) and is covered with a microporous diaphragm cap to make the readings largely independent of the liquid flow conditions. The entire stainless steel shaft of the PEROX measuring probe acts as counter-electrode. This constitutes the complete sensor part of the measuring system for hydrogen peroxide, whereas an additional reference electrode is required for peracetic acid measurements. The reference electrode type REFP-SE is used for this purpose. Thus in the standard form the H_2O_2 measurements are made with a two electrode system whereas the peracetic acid measurements are made with a three electrode system.
A special continuous electrode activation process constituting the actual know-how of this system, ensures long-term stability of the measuring system without requiring frequent recalibration.
All amperometric measurements depend strongly on the temperature, therefore we recommend temperature compensation using a Pt100 resistance thermometer probe, if temperature fluctuations occur in your application.

Applications

The environmentally compatible substance hydrogen peroxide (H_2O_2) is increasingly being used in process engineering as oxidising and reducing agent. Some examples of specific applications for continuous PEROX-H_2O_2 measurements and loop control functions are:

- Gas scrubbers, e.g. in municipal and industrial sewage treatment plant
- Ground water purification
- Drinking water oxidation
- Process water / cooling water disinfection
- Chlorine removal, e.g. in chemical processes
- Seepage water treatment in refuse dumps
- Bio-engineering
- Vat dyeing / textiles
- Swimming bath water disinfection

Peracetic acid (PAA) is frequently used as disinfectant in the foodstuff and beverage industry as well as, for example, in cosmetic, pharmaceutical and medical products. Continuous measurements and loop control are desirable whenever strict requirements have to be fulfilled for disinfection and quality control. Replenishment of the PAA-concentration in CIP-processes is a typical application for PEROX peracetic acid measurements.

3 Conditions of operation

Conditions of operation

		H_2O_2	PAA
Measuring ranges:		1 ... 20 mg/l	
		10 ... 200 mg/l	10 ... 200 mg/l
		100 ... 2000 mg/l	100 ... 2000 mg/l
		2-electrode system	3-electrode system, separate reference electrode required
pH operating range:		pH 2.5 ... 10	pH 2 ... 8 with restrictions also from pH 1 to pH 2 (please inquire)
Temperature range:		0 ... 40°C	5 ... 35 °C
		A continuous flow sample cooler is required in the case of rapid temperature fluctuations and high temperatures.	
Permissible temperature change rate:		< 0.5 K/min	< 0.5 K/min
Temperature compensation:		If ordered (see Identification Code), automatic for H_2O_2 and PAA measurements; or temperature coefficient can be set manually.	
Minimum conductivity of the sample solution up to	20 mg/l:	50 µS/cm	–
	200 mg/l:	200 µS/cm	500 µS/cm
	1000 mg/l:	500 µS/cm	2 mS/cm
	2000 mg/l:	1 mS/cm	4 mS/cm
Max. pressure:		2 bar	2 bar
Response rate of sensor, T_{90} :		approx. 20 seconds	approx. 2 minutes
Recommended sample water flow rate:		60 l/h in the DGM	
	as far as possible:	30 ... 120 l/h in the DLG	30 ... 120 l/h in the DLG
Reproducibility of the readings:		Better than 2 % of the measuring range end value	
Calibration:		When commissioning:	2-point calibration, buffer 1 and 2
		During operation:	single point calibration, buffer 2
Cross-sensitivity:		None with respect to other oxidising agents such as free chlorine	None with respect to H_2O_2
Important:		Tensides can impair the measurements. Solid material may clog the diaphragm; pre-filter if necessary. The continuous flow transmitter Type DGM can be used only for H_2O_2 measurements and is recommended only for non-turbid (clear) liquids. For all cases in which there are suspended particles in the sample solution, we recommend the continuous flow transmitter Type DLG-PER.	

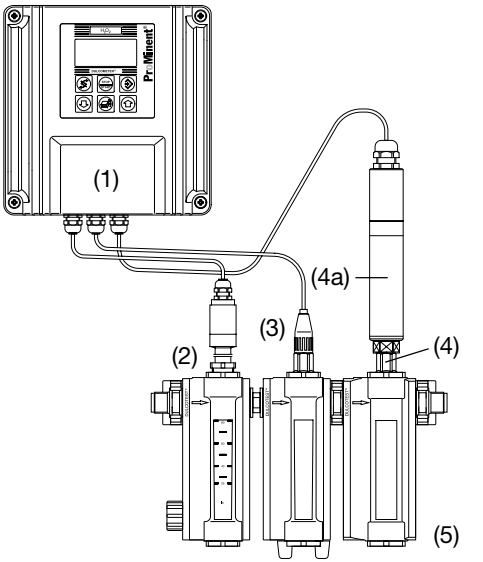
4 Set-up of the complete measuring system

Set-up of the complete measuring system

Set-up: Set-up examples

H_2O_2 measuring system in wall-mounted set-up, with automatic temperature correction and flow rate monitoring:

(1) D1C-controller Perox
(2) Continuous flow measuring probe with monitoring facility
(3) Pt100; for long-distance signal transmission, temperature signal transmitter screwed onto the Pt100
(4) H_2O_2 sensor with
(4a) signal transmitter
(5) DGM - 3 modules, alternatively
1 x DLG-PER possible too



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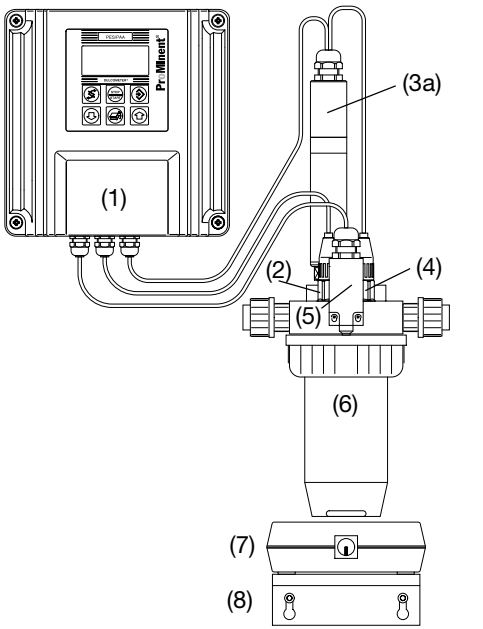
PAA measuring system in wall-mounted set-up, with automatic temperature correction and flow rate monitoring:

(1) D1C-controller Perox
(2) Pt100
(3) PAA sensor with signal transmitter (3a)
(4) reference electrode Type REFP-SE
(5) Limit switch
(6) DLG-PER probe housing
(7) Magnetic stirrer
(8) Mounting bracket



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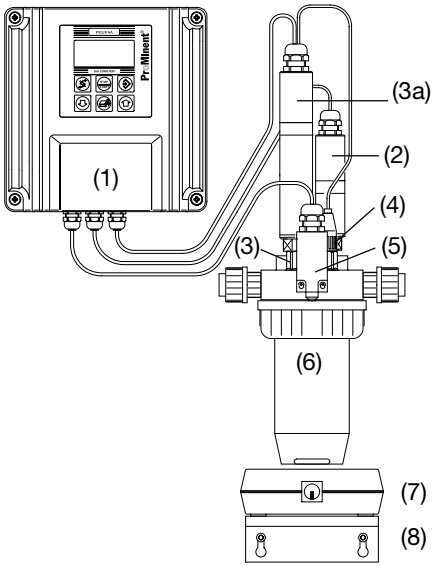
Set-up of the complete measuring system



PAA measuring system in wall-mounted set-up, with automatic temperature correction:

(1) D1C-controller PEROX
(2) Pt100 with signal transmitter
(3) PAA sensor with transmitter (3a)
(4) reference electrode REFP-SE
(5) Limit button
(6) DLG-PER
(7) Magnetic stirrer
(8) Mounting bracket

Basic equipment:	**H_2O_2**	**PAA**
D1C-controller	x	x
PEROX sensor	x	x
PEROX signal transmitter micro-mA	x	x
3-wire cable (transmitter to D1C)	x	x
Reference electrode	-	x
Continuous flow probe DGM (only for clear media)	x	-
Continuous flow probe DLG-PER	(alternative to DGM)	x
2-wire cable (for power supply switch-off)	(for DLG-PER)	x
Temperature compensation:		
Pt100 SE	x	x
Continuous flow probe module (DGM) for Pt100	x	-
Coaxial cable with SN6 plugs (for Pt100)	x	x
Temperature transmitter (for long distance between sensor and controller	(x)	(x)
2-wire cable (for temperature transmitter)	x	-
Flow rate monitoring:		
Float type flow rate meter	(for DLG-PER)	x
DGM with flow rate monitoring	x	-
2-wire cable (flow sensor connection)	x	-
Magnetic stirrer with magnetic rod	(for DLG-PER)	x
Accessories / Replacement parts:		
Replacement diaphragm cap	x	x
Polishing paste	x	x
Mounting bracket for magnetic stirrer	(for DLG-PER)	x

5 Installation

Installation

Mechanical Installation

IMPORTANT:

When installing the equipment, make quite sure that it is disconnected from the mains voltage!

IMPORTANT:

Interpose a filter in the input feed line if the liquid to be measured is turbid or dirty!

IMPORTANT:

Install a continuous flow sample cooler if the sample temperature is high or subject to large or rapid fluctuations!

The PEROX sensor is delivered with a protecting cap to protect the diaphragm and the electrode surface. Storage and transport take place in the dry state. The electrode must be polished before utilisation for the first time, in order to bring the electrode surface into a condition suitable for making measurements (see under 'Polishing the electrode'). After polishing the electrode fill the diaphragm cap completely with tapwater. Screw the sensor hand-tight into the diaphragm cap. This displaces excess water. Make serve that there are no air bubbles trapped between the diaphragm and the electrode.

Installation in DLG-PER or DGM

Just like the other probes, mount the PEROX-sensor hand-tight and leakproof in the DLG or DGM with the help of a size SW17 socket wrench. Then manually screw the transmitter(s) onto the probe(s). The power supply voltage must be in the switched-off state at the D1C while installing the signal transmitters.

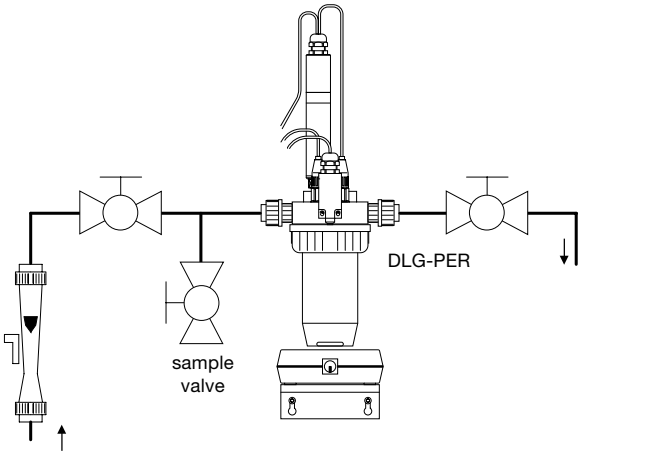
Specially for PAA
The reference electrode should be mounted in a hole adjacent, not diagonally opposite, to the PEROX-sensor.

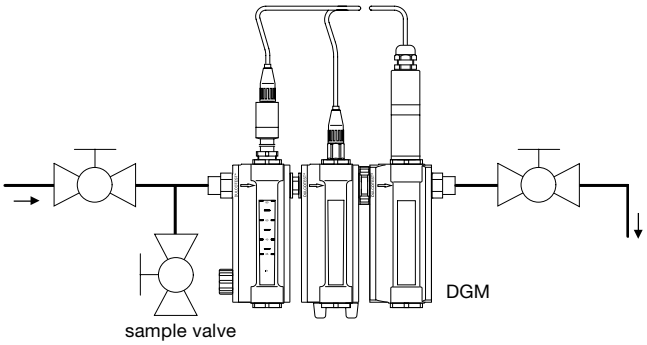
After installation of the measuring probes, the measuring cup of the DLG or DGM module should always be filled with liquid (preferably sample liquid), because **all probes except the Pt100 must be protected against dry-out**. If the probes dry out, the reference electrode could be destroyed and therefore would have to be replaced, and for the PEROX-sensor the diaphragm would have to be replaced, the electrode polished and the measuring system recalibrated.
Therefore intermediate storage of the probes must always be in the wet state kept wet.

Installation

Mounting location

Shut-off devices (e.g. ball valves) must be provided ahead of and beyond the DGM or DLG. It is advisable to install a sample-taking valve ahead of the flow indicator.





Electrical installation

Please consult the electrical installation and terminal connections diagram in the “Operating instructions D1C, Part 1” and in the “Technical data sheet for PEROX signal transmitters”.

6 Commissioning

Commissioning

SAFETY INSTRUCTIONS

Disconnect the power supply voltage every time before mounting/removing sensors, signal transmitters and other components.

The PEROX signal transmitter must not be disconnected from the PEROX sensor during operation.

The only permitted exception is during a calibration for H_2O_2 measurements in a DGM.

For the sequence of operating steps on the D1C-controller (settings, calibration, etc.), please consult the "Operating instructions D1C-PEROX, Part 2).

Functional test

Before calibrating the measuring system, make a visual inspection of all electrical connections, and check that the hydraulic connections are not leaking.

Run-in phase

Elapse of a run-in phase of at least one hour must be awaited to avoid drift of the sensor signal. Calibrate the measuring system again after 24 hours.

Calibration

After the run-in phase, the sensor must be calibrated according to the calibration menu of the D1C (PEROX) (see also Operating instructions D1C-PEROX, Part 2). Always make a two-point calibration when commencing operation for the first time. A single-point calibration with buffer 2 suffices for the recalibrations at routine intervals.
Choose the two calibration points such that the solution 1 (buffer 1) contains no PAA/H_2O_2 (zero point solution) and the solution 2 (buffer 2) corresponds to the concentration expected when making measurements subsequently. As far as possible, use solutions on the basis of the original process water for calibrating the sensor. If the process water contains components which consume PAA/H_2O_2, replace the process water with tapwater. We will be pleased to help you if you have any problems when making-up the calibrating solutions.
For calibration, the process water input feed line must be shut off and the measuring system must first be emptied, to avoid any mixing of calibration solution and process water.

Calibration in the DLG-PER:

- Switch to the calibration menu according to "D1C-PEROX Part 2".
- Close the shut-off valves.
- Empty the process water out of the measuring system.
- Empty the cup of the DLG and then fill it with calibrating solution.
- Screw the cup back into the DLG.
- Switch-on the magnetic stirrer to feed liquid to the sensor.
- Proceed according to the calibration menu "D1C PEROX Part 2".
- When changing the calibrating solutions, rinse the cup and the probe thoroughly with "buffer 2", empty the cup and then fill it with buffer 2.
- Continue according to the calibration menu "D1C PEROX Part 2".

Calibration in the DGM (only for H_2O_2)

By sample-taking procedure

- Switch to the calibration menu according to “D1C PEROX Part 2”.
- Shut-off the process water input feed line.
- Empty the process water out of the measuring system.
- Fill the DGM with buffer solution via the sample-taking valve.
- Make sure that adequate amounts of liquid flow past the sensor, either by pumping between the outflow and the sample-taking valve or by feeding-in “buffer” solution as long as the calibration is running.
- Proceed according to the calibration menu “D1C PEROX Part 2”.
- When changing the buffer solutions, empty out the first buffer solution, then rinse thoroughly with “buffer solution 2”.
- Continue according to the calibration menu “D1C PEROX Part 2”.

By taking-out the sensor/transmitter assembly (in this exceptional case leave the controller switched-on, although the PEROX transmitter is disconnected from the sensor)

- Switch to the calibration menu according to “D1C PEROX Part 2”.
- Shut-off the process water input feed line.
- Disconnect the PEROX transmitter from the sensor.
- Unscrew the sensor out of the DGM.
- Connect together the sensor and the transmitter again.
- Then carry out the calibration in a beaker filled with the respective buffer solution. Thereby stir the liquid in the beaker with the sensor to make the liquid flow past the sensor or use a magnetic stirrer.
- Proceed according to the calibration menu “D1C PEROX Part 2”.
- To change the calibrating solutions, rinse the beaker thoroughly with buffer 2, empty it and then fill it with buffer 2.
- Continue according to the calibration menu “D1C PEROX Part 2”.

Calibration intervals

The proper calibration intervals depend strongly on the process conditions and the substances actually present in the process water, therefore no generally valid specifications can be given. Under laboratory conditions (pure aqueous solutions of H_2O_2) the calibrations intervals are about 3 months. To determine the optimum calibration intervals under process conditions, determine the PAA/H_2O_2 concentrations by independent methods (e.g. titration) at regular intervals (e.g. initially daily). (Note: If easily oxidised substances are present, titration with potassium permanganate is not possible.)

For definite results, the sample-taking point must lie in the immediate vicinity of the sensor (see under “Mounting location”). If this condition is not fulfilled, there may be considerable discrepancies between the sensor reading and the externally determined PAA/H_2O_2 content of the sample.

7 Operation

Operation

IMPORTANT!

The PEROX system should always be left switched-on, even when the process which is monitored/controlled is temporarily shut-down (e.g. over a weekend). Otherwise the sensor would lose its proper surface condition which is important for making measurements. When switching-on again after switch-off, the sensor is ready again for making measurements only after elapse of a run-in time. The measuring cup of the DLG or DGM must always be kept filled with process water or tapwater (see above).

Maintenance

It is advisable to make a regular visual check of the sensor condition, in particular the diaphragm. If the diaphragm is contaminated with dirt, replace the complete diaphragm cap.
If the electrode surface is dull, polish the electrode and replace the diaphragm cap.

Diaphragm replacement

- Disconnect the PEROX system from the mains voltage.
- Close the shut-off valves.
- Unscrew the transmitter from the sensor.
- Screw-out the PEROX sensor from the DLG/DGM.
- Unscrew and detach the diaphragm cap.
  Hereby check the condition of the electrode surface. If the platinum or gold electrode appears dull or modified, polish it (see “Polishing the electrode”).
- Fill new diaphragm caps completely with tapwater.
- Screw the sensor hand-tight into the diaphragm cap. This displaces excess water. Make sure that there are no air bubbles trapped between the diaphragm and the electrode.
- The diaphragm should be slightly convex with respect to the exterior.
- Screw the sensor leakproof into the DLG/DGM filled with water or process water (SW17 wrench). Screw-on the signal transmitter (SW22 wrench). Establish the signal connection to the D1C.
- Open the shut-off valves.
- Switch-on the mains voltage.
- Recalibrate

After replacing the diaphragm without polishing the electrode, the sensor **does not** require a run-in time, therefore calibration is possible after a short time.
After polishing and diaphragm replacement, we recommend a 2-point calibration (buffer 1 and 2), whereas a regular slope correction (buffer 2 only) suffices in routine operation.

Polishing the electrode surface

- Put a pea-size amount of polishing paste onto a soft (paper) rag.
- Press the electrode into the polishing paste and turn it under gentle pressure.
- Completely remove the polishing paste from the electrode and sensor shaft by rinsing under lukewarm running water.
- The surface of the platinum or gold electrode must have a metallic gloss again. If not, repeat the polishing process.
- Do not touch the electrode surface again. Contamination with skin grease or sweat would impair the sensor function.

For further procedure steps, see under “Diaphragm replacement”.

8 Spare Parts and Consumer Material

After polishing the electrode, the surface must be brought again into the condition required for making measurements. This requires a run-in time of at least 1 hour. Calibration can be carried out thereafter. Repeat the calibration after 24 hours.

Spare parts and Consumer Material:

H_2O_2 sensor shaft, Type H2.10 P, complete with diaphragm cap	792976
Corresponding signal transmitter, Type PEROX-micro-H 1.20-mA	741129
H_2O_2 replacement diaphragm cap, Type M 2.0P	792978
PAA sensor shaft, Type P2.10B, complete with diaphragm cap	809150
Corresponding signal transmitter, Type PEROX-micro-P 1.30-mA	741128
PAA replacement diaphragm cap, Type M 2.0 B	809154
Reference electrode, Type REFP-SE	1000505
Temperature sensor, Type PT 100 SE	305063
Temperature transmitter, Type PT 100 V1	809128
Set for continuous flow probe module (triple) (hose connectors 8 x 5)	DGMA 320T000
Set for continuous flow probe module (triple) (screw-fittings d16/DN10)	DGMA 320T010
Inline Probe Housing, Type DLG-PER (including limit button with 2 normally open contacts)	1000165
Magnetic stirrer for 100-240 V AC/50-60 Hz	790915
Magnetic stirring rod 15 x 6 PTFE	790917
Mounting bracket for magnetic stirrer, PVC	100166
Polishing paste for PEROX sensor, 90g tube	559810
Connecting cable D1C - PEROX transmitter, Type LiYY 3 x 0.25	791948
Connecting cable D1C - Temperature transmitter / limit button 2 x 0.25	725122
Coaxial cable D1C to temperature sensor SN6-open, 2 m	305030
Coaxial cable D1C to temperature sensor SN6 open, 5 m	305039

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