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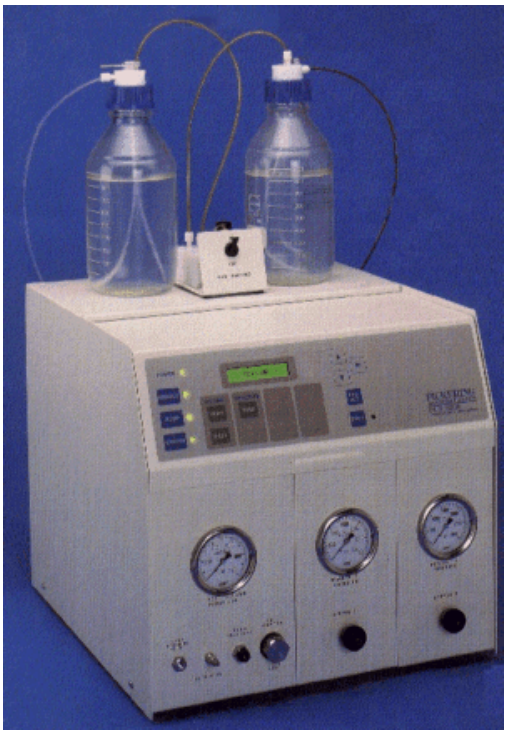
Technique of Post-Column Derivatisation (PCD)

As already mentioned on the page entitled "Getting into Post-column Derivatisation", only a few components are *theoretically* required to build a post-column derivatisation system.

For a reliable, easy to use system, however, a lot more needs to be considered as you can see from the Pickering's **PCX5200**!



PCX5200

The Pickering's **PCX5200** by has various construction characteristics that make the post-column derivatisation technique more reliable and robust. In addition, safety devices protect the system from being damaged during routine works.

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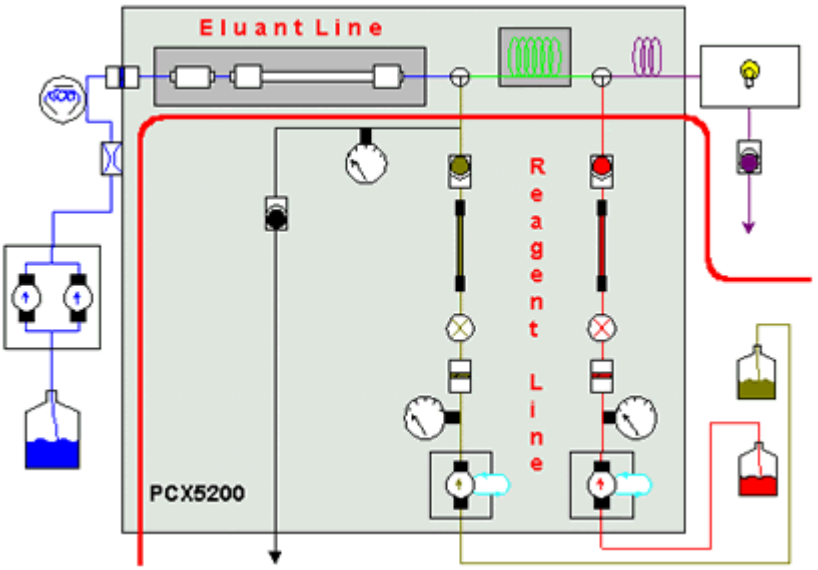

*Flow diagram of the 2-step **PCX5200***

You will find details about Materials Used and Technical Data at the end of this chapter.

The following functions and components, respectively, are integrated in the **PCX5200**:

- Reagent Delivery
- Thermostatting of the Reactor
- Thermostatting of the Analytical Column
- Safety Features

Reagent Delivery

The addition of the reagent solution should take place as pulse-free as possible!

Each pressure change in the detector cell causes a variation of the detector base line. The signal-to-noise ratio becomes more unfavourable and, consequently, the sensitivity of the method decreases.

In the **PCX5200** model**,** robust, reliable single-piston pumps are used for the reagent delivery. The flow rate is controlled via the stroke volume. A standard piston seal wash extends the life of the piston seal. Highly *efficient* pulsation dampening is achieved by the installation of a manometer and a restrictor behind the pump.

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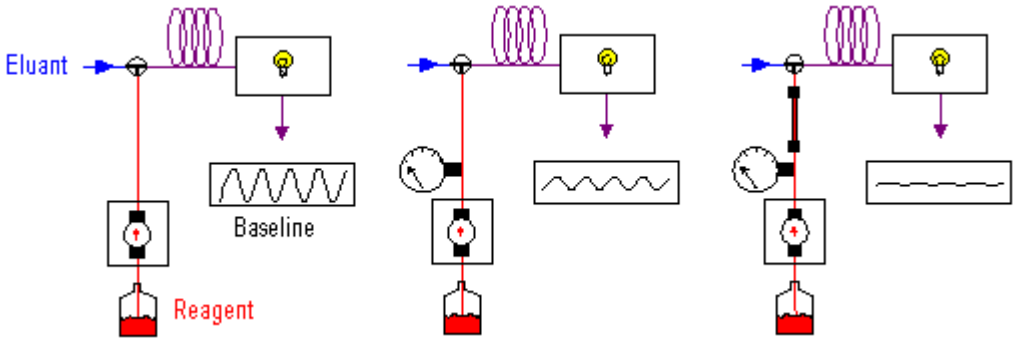
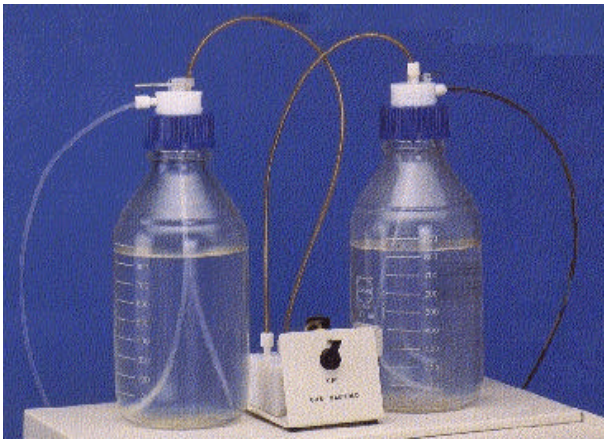


Pulsation dampening of the reagent pump by installation of a manometer (pulsation dampener) and a restrictor (column)

The manometer gauge (Bourdon-gauge) functions as the pulsation dampener, which becomes only fully functional by means of the backpressure of the column (restrictor). The working order can be compared to a reservoir and dam, used to regulate a river niveau.

Components of the Reagent Line:

1l-Reagent Bottles: these are plastic coated, as they are exposed to overpressure (ca. 0,5 bar inert gas). Consequently, the life of oxygen-sensitive reagents is extended (e.g. *o*-phthalic dialdehyde-solution) and the suction of the reagent pump is enhanced. Gas and reagent lines are made from highly oxygen proof Saranâ . For the reagent line, a stopcock is integrated in the bottle top.



Gas diffuser block and reagent bottle

Manometer (0 – 3000 psi): beside pulse dampening, it is used for pressure control of the reagent line.

Bypass-Valve: used for rinsing the reagent line, the pump head and the manometer gauge. In combination with the stopcock of the reagent line, air bubbles are removed very efficiently.

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Inline-Filter (2µm)

Restrictor : available in different strengths depending on the viscosity of the reagent solution. Indispensable for pulsation dampening.

Check Valve (60 psi): prevents that the gas pressure pushes the reagent solution from the reagent bottles into the "standing" main line when the HPLC is switched off, or that the mobile solvent flows into the "standing" reagent lines when switching the HPLC on. Both occurrences can lead to serious system damages.

All components between pump and T-piece are assembled on a bearing plate as "Flow Conditioner" and easily accessible.

"Flow Conditioner"

Further Components of the Pickering-System

"Post-column" Manometer (0 – 1000 psi): assists the control of pressure post column. This parameter is an important indicator for the operating state of the Pickering-system. The post-column pressure is composed of the flow rates of HPLC and reagent pump(s) and of the backpressures of the reactor, detector cell and backpressure regulator. Deviations could result from leakage, clogging or a wrong flow rate. In addition, the post-column pressure may only rhythmically pulsate around +/- 5 psi due to the excellent pulsation dampening.

Mixing-T-Piece: is a T-piece! The geometry of the connections has no influence on the mixing grade, if specific recommended parameters (total flow rate and bore hole diameter) are being adhered to.

Backpressure Regulator (40 – 100 psi): suppresses the boiling of the solutions in the hot reactor and a possible formation of bubbles in the detector cell.

Precolumn-Inline-Filter (0,5µm)

Thermostatting of the Reactor

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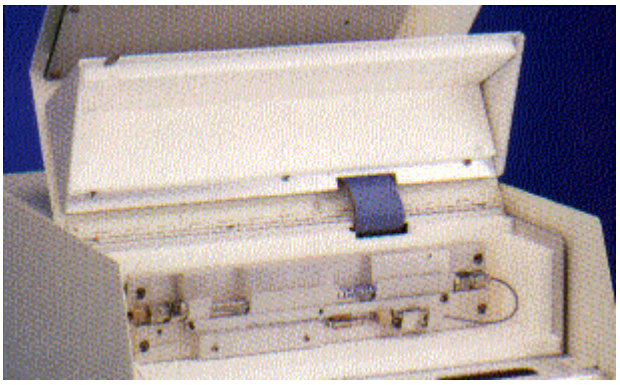
A constant reactor temperature is important for the reproducibility of the derivatisation!

The *heatable* reactor (RT+10°C to 130°C) consists of a PTFE-capillary of suitable length (up to 1.4 mls volume), which is wound round a heating block insulated with glass wool. The narrow inside diameter (0.28mm) minimises peak broadening and, at the same time, the larger wall thickness enhances the robustness of the reactor. Consequently, this type of reactor is still stable at 130°C and up to 42 bar.
At larger volumes (up to 3.0 mls), capillaries are used with a wider inside diameter (0.5 mm). In such a case, however, the capillaries are "crocheted", since the laminar flow may prevent adequate mixing of the eluant with the reagent solution.

Thermostatting of the Analytical Column

The *column oven* is integrated in the PCX5200 to guarantee the modular structure of the whole system (HPLC and **PCX5200**). The easy accessible column oven fits columns of up to 25 cm length and 8 mm diameter as well as a separate precolumn. The temperature ranges from RT+5°C up to 75°C.



Column Oven

Safety Devices

Pressure Relief Valve (35 bar): prevents bursting of the reactor or detector cell should the capillaries become clogged by leading-off the eluant/reagent stream into the waste.

Pressure Sensor: most derivatisation reagents immediately damage the column material as soon as they enter the column. This back flow of reagent solution occurs, if the "post-column" pressure (typically 10 to 20 bars) exceeds the "precolumn" pressure. In order to prevent this, a pressure sensor is installed between the HPLC pump and the injector. The **PCX5200** can be activated only if the "precolumn" pressure exceeds 35 bar. However, the **PCX5200** completely switches off, if the pressure is less than 35 bar (reactor heating and reagent pumps). Then, the system can be only be restarted manually after control by the user.

Temperature safety: the column oven and the reactor are safeguarded against exceeding the maximal temperature.

Inert PCX5200

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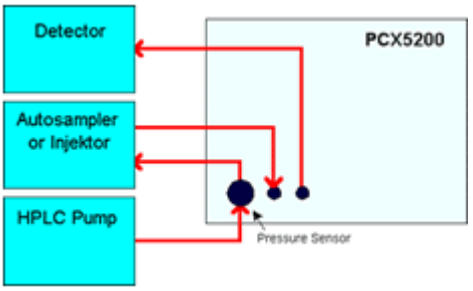
For applications with particularly corrosive reagent solutions (e.g. methanolic sulphuric acid), the use of the *inert* **PCX5200** model *is* recommended. All parts exposed to the corrosive reagent solution are designed accordingly. Also, see under Materials Used.

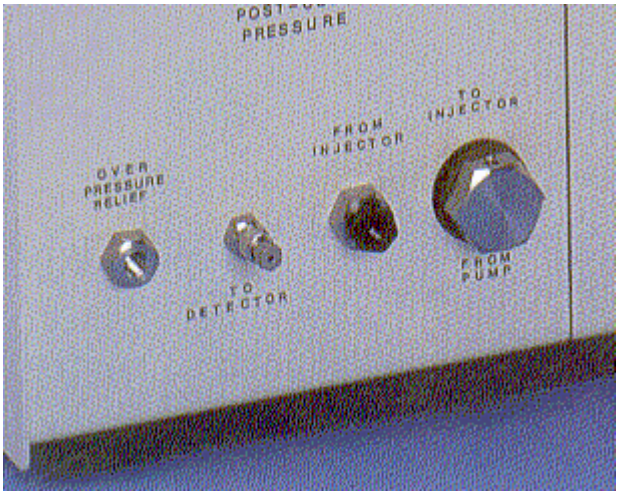
Installation of a PCX5200

The installation of the **PCX5200** is very easy. In the current HPLC-system, the **PCX5200** is installed between the pump/injector and the detector. Fittings and ferrules needed for the connection on the side of the **PCX5200** are included with the delivery.



*Installation diagram for the **PCX5200***

All connections are easily accessible at the front of the **PCX5200.**



Connections

Conversion for other Applications

For alternative applications, it is possible that a heatable reactor with a different volume is required in the **PCX5200**, this is done easily and quickly. The exchange of the heatable reactor takes less than three minutes and can be carried out without tools. Only two "fingertight"-fittings and an electric jack plug must be loosened and then tightened up again.

The restrictor in the reagent line needs to be exchanged, if a reagent, which is not dissolved in water, methanol or a solvent of similar viscosity, is utilised.

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When the Technique of Post-Column Derivatisation isn't quite so robust,....

The installation of a **PXC5200** is always conducted by technically experienced personnel. As part of the installation procedure, potential technical issues or problems, and appropriate solutions will be discussed with the user. A very detailed and informative handbook also helps to solve problems. LC*TECH* additionally offers a Service Contract.

Electronic

The **PXC5200** microprocessor control allows five sets of parameters from different applications to be saved. The RS232-interface enables monitoring of the column oven and/or reactor temperatures as well as the external control of the **PXC5200** using a PC (software supply by the user).

Materials Used

The following materials come into contact with eluants and the reagent solution.

PCX5200:

316 high-grade steel	17-4 PH high-grade steel
Teflon FEP	Saran PVDC
Hastelloy-C	Kel-F CTFE
PEEK	Viton
Borosilicate glass	Sapphire
UHMW Polyethylene	

Inert **PCX5200**:

316 high-grade steel	17-4 PH high-grade steel
Teflon FEP	Saran PVDC
Kel-F CTFE	PEEK
Tefzel ETFE	Borosilicate glass
Sapphire	UHMW Polyethylene

Technical Data

Dimensions:	37 cm x 38 cm x 38 cm (H x B x D)
Weight:	13 to 15 kg depending on configuration (net)
Current:	200 - 240 Volts; 50/60 Hz; 0,8 A; 200 Watts

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Reagent Pump(s):	Flow rate 0,05 – 0,7 mls/min
	Stroke frequency 25 Hz
	Delivery pressure to 410 bar
	Sapphire piston
	SS316 high-grade steel pump heads (with PEEK inserts for the inert model)
	Manual piston seal wash
Heatable Reactor:	0,15 mls – 3,0 mls (any intermediate sizes available)
	Room temperature + 10°C to 130°C
	Temperature constancy ± 0,4°C
	Protected against uncontrolled heating up (150°C)
Column Oven:	Room temperature + 5°C to 75°C
	Temperature constancy ± 0,4°C
	Protected against uncontrolled heating up (80°C)
	Columns up to 25 cm long and 8 mm diameter

Gas supply for up to four bottles including pilot-controlled reducing valve (inlet pressure 1 – 6 bar, end pressure 4 – 6 psi), stopcock and pressure relief valve (8 psi).

Reagent bottles with Saranâ -tubing and plastic covering approved for overpressure of up to 1 bar.

Microprocessor control of temperatures. *RS232-interface* for data monitoring and/or control of the system by PC (software supply by the user).

Questions or Comments?

If you have questions or comments, then please don't hesitate to send us an e-mail: inf@ingenieria-analitica.com