

Operation Manual for PD3100 Post-column Derivation Statement

V1.0.6

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Please read the manual carefully before operation.

1. Overview

Post-column derivation, also called post-column derivation reaction, is a process of converting some compounds without detective characteristics into those with detective characteristics (derivatives). During the HPLC analysis, a post-column derivation instrument is on the basic of the technology to obtain easily detected compounds via chemical reaction after separation. For example, by some kind of chemical reaction, compounds without UV absorption or fluorescence properties can be converted to derivatives with UV absorption or fluorescence properties, whose detective sensitivity maybe increase by orders of magnitude.

Post-column derivation mixes derivatization reagent and substances from column together. Then the mixture flows to a reactor to finish derivative reaction within enough time. If the reaction rate is low, it can be improved by heating. Some other reaction need two or more derivatization reagent in turn.

2. Introduction

PD3100 post-column derivation is developed and produced by Dalian Elite Analytical Instrument Co., Ltd., using update design concepts, with all flow path made by nonmetallic material, precise temperature controlling, terse and beautiful external appearance. It can be connected with any LC of different branch, ensuring high-efficient, accurate and easy processing.



Figure 1 Design sketch

3. Structure

PD3100 post-column derivation is composed of four parts: double-reactor, temperature control, fluid cooler and circuit control.

Capillary made of PEEK is in the reactor. Fluid flows from column and finish derivative reaction with derivation reagent in the reactor. If necessary, the reactor has temperature-rising control ability.

Omron E5CC versatile controller is selected to realize temperature control. To prevent temperature beyond control for uncontrollable reasons, safeguard of ultratemperature is designed and installed in PD3100 post-column derivation. When temperature is higher than 160 °C, the instrument will automatically power off. After it cools down, the temperature will automatically warm up.

If the derivation reaction has to proceed at 100 °C or more, cooling measures for the fluid from reactor must be taken. Otherwise the detector might be burnt out. Fluid cooler system of PD3100 post-column derivation reactor helps users operate without concern.

4. Technical index

4.1 Functional parameters

PD3100 is a product combined double-derivative reaction module, double independent precise temperature control module, and fluid cooler.

Modular design of derivation makes simple replacement of components.

All fluid path is made of insert materials. Materials contacting with liquid are PEEK, which have good biocompatibility and widespread application.

Independent temperature control, automatic intelligent temperature regulation, can cut off automatically, with over-heating protection circuit.

Widespread chemical adaptability, will not be influenced by acid, base, organic and surfactant.

pH Range: 0~14.

4.2 Performance index

Temperature control range of derivation reaction module: RT+3°C~155°C.

Accuracy of temperature control: $\pm 0.1^{\circ}\text{C}$.

Precision of temperature control: $\pm 0.2^{\circ}\text{C}$.

Stabilization time: 15min (150°C below).

Volume of derivation reaction module:

The 1st level of derivation module is 0.3mL,

The 2nd level of derivation module is 0.5mL.

Maximum pressure endurance of derivation reaction module:

10 MPa .

Power supply: AC220V, 50 Hz, 320W.

Dimension: 420*260*100 mm (length*width*height)

5. Accessories

Item	Quantity
PD3100 post-column derivation	1 pc
Power cable	1 pc
Fuse	1 pc
PEEK fingertight fitting	8 pcs
PEEK union	2 pcs
PEEK tubing (1/16" OD*0.007" ID)	2 m



6. Connecting tubing

Use 1/16" OD*0.007" ID PEEK tubing and fingertight fittings to connect column and PD3100 post-column derivation "Column" interface.

Use 1/16" OD*0.007" ID PEEK tubing and fingertight fittings to connect PD3100 post-column derivation "Outlet" interface and detector.

Two kinds of derivatization reagent flowing from pumps connect to PD3100 "Inlet1" and "Inlet2" interface respectively. 1/16" OD*0.007" ID PEEK tubing and fingertight fittings are commended here.

7. Operation

Item	Function
Temp. Controller1	To press up/down key  and  to set the reaction temperature of the reactor
Temp. Controller2	
Column	Entry of post-column derivation
Inlet1	Entry of derivatization reagent1 pump
Inlet2	Entry of derivatization reagent2 pump
Outlet	To connect to flow cell entry

8. Solvent perfusion and solvent exchange

Make sure the PD3100 post-column derivation is properly installed, and the tubing between column and detector is connected correctly.

Choose solvent reservoir and fill in mobile phase according to analytical conditions.

- a. First start LC high pressure constant flow pump, and set flow rate. Disconnect “Outlet” interface and detector, and stop the pump when liquid flowing stably from tubing.
- b. Start the pump for derivation reagent1, and stop it when liquid flowing stably from tubing.
- c. Start the pump for derivation reagent2, and stop it when liquid flowing stably from tubing.
- d. Reconnect the “Outlet” interface and detector to start analysis.

During the above operation, notice totally eliminating air in tubing from beginning to end.

9. Power off

Please turn off the power, after using PD3100 post-column derivation. It is necessary to avoid crystallization and precipitation in the system when shutting down.

- a. Short-term shutdown (for overnight or weekends)

Notice the precautionary measures of dangerous solvent or the solvents which can form precipitation or by-products.

Purge away harmful mobile phase of PD3100 post-column derivation and other instruments.

Flush the column according to Columns Handling Instructions. Flush and remove salt of buffer solution. Otherwise, residual salt will crystallize after mobile phase evaporating.

Remove chloroform or some other solvents which can decompose into hydrochloric acid.

If weekend shutdown, 50/50 methanol/water (V/V) is suggested to flush PD3100 post-column derivation, high pressure constant flow pump, column and detector.

- b. Long-term shutdown

The steps are the same as short-term shutdown.

Unload the column, and purge PD3100 post-column derivation with water first and isopropanol then.

Shut down PD3100, and disconnect all the tubing. Block off all the outlets of PD3100 post-column derivation.

PD3100 must be hold in clean and dry place.

If using PD3100 again, purge it thoroughly with appropriate column solvent before being connected to column and starting the system.

10. Maintenance

Follow standard lab cleaning practices.

Use high purity solvent (chromatogram pure) as mobile phase. It is necessary to use chromatogram class purified water or filtered deionized water.

All solvents must be filtered by 0.45 μ m filter membrane, to prevent solid particle blocking the tubing.

Make sure stainless steel tubing has been passivated and flushed before being connected to pump.

Clean up spills near or on the instruments right away in proper way. The spills may damage the instruments surface and functions.

11. Cautions

Please pay particular attention to treat flammable solvent. Leakage is strictly prohibited. Make sure the exhaust gas is not harmful.

During the analysis, chromatography class pure mobile phase, or analytically pure at least, is asked to use.

The cutting surface of PEEK tubing cut by specialized tubing cutters is asked to be leveling and smooth. Otherwise solutions and gas tend to leak.

The instrument damage caused by buffer salts precipitation in the connecting tubing is outside the scope of free warranty.

The first display screen shows the actual temperature, and the second shows the setting temperature. Yellow indicator lights means it is heating.

Purified water is saved in the tubing of PD3100 post-column derivation before leaving the factory. Please pay attention that the LC mobile phase miscible with water.

Except for special instructions, the instrument does not have explosion-proof function.

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