

PLE™ Extraction System

Instructions Manual

FMS

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WARNINGS / CAUTIONS

Important: Do not reuse the PLE column teflon end caps more than twice.

The PLE must be used according to this manual, otherwise safety may be impaired.

Unplug all power cables from the mains outlet before servicing the PLE.

Unplug all Heater/Fans power cables from the mains outlet before unplugging from the PLE.

1. The PLE is flexible, yet highly sensitive instrument. Read this User's Manual before use.
2. Only adequately trained personnel should operate the system. A documented training program based on training received during the installation/training session from installation engineers is highly recommended
3. Do not shut off the system main power switch, the computer or individual module power switches while the system is running.
4. Do not connect or disconnect connector (s) while the system power is on.

5. Do not remove or install module (s) while the system power is on.
6. Always, replace fuses with the same type and value to protect against fire hazard.
7. Always operate the system under specified electrical specifications.
8. Always operate the system with the safety shields installed.
9. Always wear protective eyewear and gloves when operating the system.
Do not use powdered gloves.
10. Always observe safe laboratory practices and rules when operating the system.
11. When handling solvents, samples and other chemicals, changing tubing, or operating the system, always observe good laboratory practices.
12. When using the system, follow generally accepted procedures for quality control and methods development.
13. When handling solvents changing columns or other components, and during operation, always observe good laboratory practices. Know the physical and chemical properties of solvents and refer to the Material Safety Data Sheets for appropriate handling precautions.
14. Use of strong HCL will damage the system and could be hazardous to the operator and will void the system warranty.
15. Do not loosen fittings while the system is running.
16. To prevent the loss of samples, make sure sufficient solvent exists for the run.
17. Replace fraction bottles in a timely fashion, to prevent spill over
18. Use of columns other than FMS disposable columns voids all warranties.

19. Changes or modifications to this system without written consent from the manufacturer may void warranties.
20. Do not nick, kink, or bend sharply tubings. This may restrict flow or cause tubing to fail.
21. Do not touch any moving parts while the system power is on.
22. Do not touch the inside of the heater blocks. Always open and close the heater blocks by the Teflon handles to prevent burns.

For technical help please contact:

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Specifications:

Pump Flow Rates0.1 to 35.0 mL/min
Pump Pressure0 to 2,000 psi
Heater block Temp.....Room temp to 150 C
Dimensions34" (86cm) high x 56" (142cm) wide x 19" (48cm) deep
Weightup to 400 lb (181kg)
Power120 VAC/ 60 Hz; or 230 VAC/50
Power consumption.....PLE control module: 100watts to 400watts
 10" heater block set of two: 1KW
 (110VAC at 9amps)
 5" heater block set of two: 500watts
 (110VAC at 4.5amps)

Accessories.....-5” heater block set
-10” heater block set
-5ml to 100ml cells with Teflon or
Stainless steel end caps vertically oriented
With flow from bottom to top
Pneumatic requirements... N2 at 30psi
Control.....PIO from Pentium PC, windows XP

1. Installation

1.1 Unpacking and Inspection

Prior to opening the shipping containers, inspect it for damage or evidence of mishandling. If it has been damaged or mishandled, notify the manufacturer before opening the container. Once the container is opened, inspect the contents for damage. Any damage should be reported to the manufacturer immediately. Save the shipping container. Check the contents against the packing list.

1.2 Location/Environment

The preferred environment for the PLE is normal laboratory conditions. The area should be clean and contaminant free. If the instrument is not mounted on the PLE cart it should be located on a stable flat surface with surrounding space for ventilation and the necessary electrical and N2 connections.

1.3 Electrical Connections

Unpack the PLE and check the voltage setting on the Power Entry Module on the back of the PLE control module. Make certain the voltage setting agrees with the power to be supplied to the unit. A system which is connected to a 100-120Vac voltage source should have a voltage setting of “110VAC”, and a system connected to a 220-240Vac voltage source should have a voltage setting of “220VAC”.

If the voltage setting is correct, plug the PLE control module into a properly grounded electrical outlet, and plug the PLE heater block power cables into separate grounded electrical outlets.

Each two sets of 10” PLE heater blocks must be plugged into separate grounded electrical outlets rated at 20amps, and three sets of 5” PLE heater blocks must be plugged into separate grounded electrical outlets rated at 20amps.

WARNING: Do not bypass the safety ground connection as a serious shock hazard could result.

If the PLE control module does not have the correct voltage configuration, notify the manufacturer or your local distributor.

1.4 Other Connections

Connect the 50 pin gray flat cable from the computer to the 50 pin connector in the rear of the

PLE control module marked “PC”.

All other connections must be firmly in place and if necessary screwed in.

1.5 Plumbing connections & pump priming

1.5.1 Screw the 10-32 fitting end of the “main transducer tubing” into the output port of the main pressure transducer and tighten using a wrench.

1.5.2 Connect the female quick connect fittings on the “main transducer tubing” for each channel to the male quick connect fitting of the input valve for each channel.

1.5.3 The PLE pump must be primed before use. Draw some solvent into the priming syringe and inject into the pump inlet tubing to fill the pump.

2. Power up

2.1 PC power up & set up

2.1.1 Boot up the PC.

2.1.2 Launch the DMS6000 software

2.1.3 Open six temperature log files and six pressure log files

2.1.4 Open a PLE program

Please refer to the PLE DMS6000 software operator's manual for instructions on the PLE software.

2.2 PLE power up & set up

2.2.1 Turn the PLE extraction module power switches on

2.2.2 Turn the PLE control module power switch on

2.2.3 Run the PLE program and stop it as soon as allowed

2.2.4 Connect the PLE nitrogen line to a nitrogen tank (with a 1/4"NPT outlet). The nitrogen

tank must be set at 25-35 PSI.

2.2.5 Immerse the PLE pump inlet tubing in a bottle full of solvent & cover with aluminum foil.

3. Construction

The PLE plumbing and wetted parts are constructed from stainless steel and Teflon or Teflon based materials, making it compatible with all organic solvents and most non organic chemicals.

The materials of construction make the PLE easy to clean. However, care must be taken to keep the PLE contaminant free. This is accomplished by running wash programs after every sample run.

The PLE is constructed of modules and cabinets.

3.1 Cabinets

3.1.1 The PLE small cabinet holds the PLE control module and the PLE pump module.

3.1.2 The PLE large cabinet holds the PLE extraction module and the PLE column module, as well as external PC boards and interconnection cables.

3.2 Computer

The PLE computer includes a PIO card supplying the IO signals necessary for the operation of the PLE.

It also includes a 2 channel A/D card for translating the temperature probe and the pressure transducer signals to digital format to be displayed on the PC screen.

3.3 Modules

3.3.1 PLE control module

The PLE control module communicates with the PLE computer, PLE extraction modules, and the PLE pump module.

It receives PIO signals from the PLE computer to control valves, PLE pump, and heater blocks.

It sends PIO signals to open and close valves to the PLE extraction modules.

It processes the PIO signal to control the PLE pump and sends the processed signal to the PLE pump module.

It processes the PIO signals to control the heater blocks and sends the processed signals to the PLE column module.

It receives temperature probe and pressure transducer signals from every channels and sends them to the PLE computer for processing and plotting.

3.3.2 PLE Pump module

The PLE pump module receives start/stop signals from the PLE control module.

For further information on this module refer to the “PLE pump module instruction manual” section and appendix C of this manual.

3.3.3 PLE extraction module

The PLE extraction module contains the main plumbing, input valve, output valve, Nitrogen valve, channel pressure transducer, mechanical pressure gauge, manual isolation valve, and pressure relief valve.

It receives the PIO signals to control the input, output, and nitrogen valves from the PLE computer through the PLE control module which are then processed to control these valves.

It sends the channel pressure transducer signals to the PLE computer through the PLE control module for further processing and eventual display on the computer screen.

3.3.4 PLE column module

The PLE column module contains the high pressure toggle clamp, the column holding cups, the cooling fans, the heater blocks, and the temperature probe.

It receives the signals to control the fans and the heater blocks from the PLE control module.

It sends temperature probe signals to the PLE computer through the PLE control module for further processing and eventual display on the computer screen.

4. Operation

The PLE (Pressurized Liquid Extraction) system uses high pressure of up to 2500psi and high temperature of up to 200 degrees C to do extraction of different analytes from a wide variety of sample; such as food, environmental, agricultural products, pharmaceuticals, etc.

WARNING: Due to the operational characteristics of the PLE, i.e. High pressure and high temperature, extra care must be taken to operate the system according to the specifications and instructions in this manual.

After sample has been loaded into the PLE column, it is placed in the PLE column holding cups and clamped down. The clamping pressure must be adjusted according to the instructions of the engineer doing the installation and training.

The safety shield must then be installed on each and every channel.

The PLE then fills every column (sequentially) with solvent. The columns are pressurized. Under pressure the columns are heated for a certain period of time, in which extraction takes place.

The columns are pressurized before heating to prevent the solvent from boiling.

During the extraction step, the heating would tend to increase the pressure inside the PLE column. the pressure relief valve, which is set to a certain pressure will open slightly to relieve

the pressure and thereby maintain it at the desired pressure. This pressure maintenance will release small amounts of the extract into the fraction vessel.

Some components used to construct the PLE (valves etc.) have a temperature rating of 70 degrees C. to prevent hot solvent from being released into the system plumbing, the columns are cooled before depressurization.

The cooling step activates the cooling fans to cool the PLE column. This would prevent the boiling of the solvent when the system is depressurized. Boiling of the solvent extract would not only damage the system but cause the loss of semi volatile target compounds. During the cooling step the inlet and outlet valves are kept closed to isolate each channel from the others.

After the cooling step the depressurization step is executed, in which all channels are depressurized and the extracts are released into the fraction vessels. This is done by opening the outlet valves for all channels.

The inlet valves should still be kept closed to assure the channel isolation and prevent possible back flow of solvent to cause cross contamination.

The next step in the extraction process is to flush the PLE columns with solvent to push the extract out into the fraction vessels. These steps are identical to the column filling steps.

Depending on the application and method one, two, or more cycles maybe needed to complete the extraction.

Regardless of the number of extraction cycles, the last step of the program (not every cycle) flushes each PLE columns (sequentially) with nitrogen to get the remainder of the extract out into the fraction vessel and dry the columns as much as possible.

The nitrogen valves are then closed.

4.1 How to do extraction on the PLE:

4.1.1 Turn the PLE computer and the PLE system on.

4.1.2 Take PLE columns out of vacuum sealed bags. Take one Teflon end cap off each one and place on a clean sheet of aluminum foil.

4.1.3 Pour some Na₂SO₄ or hydromatrix in each PLE column (the amount to be determined by the resident chemist).

4.1.4 Pour the prepared sample into the column, and spike it.

Note: The sample is either dry (by freeze drying or other drying technique) or raw. Either way it has to be mixed with Na₂SO₄ or hydromatrix in a clean glass beaker until it is free flowing.

4.1.5 Fill each column with Na₂SO₄ or hydromatrix.

WARNING: Please make sure to fill the column to right below the counter bore line. The counter bore is as far as the Teflon end cap must travel in order to make a seal, so if you fill above that line the seal will not be made and solvent leaks will result.

4.1.6 Replace the Teflon end cap into the column.

4.1.7 Label each column with its sample number, place on the proper PLE channel, and clamp all the way down.

Note: The clamping down and the clamping force must be done according to the instructions given by the installation and training engineer.

4.1.8 Replace the safety shield.

4.9 Label the fraction vessels.

4.1.9 Place the fraction tubing (PLE outlet tubing) in the fraction vessel, and cover with aluminum foil.

4.1.10 Place the PLE pump inlet tubing in a clean bottle of solvent and cover with aluminum foil.

Note: The appropriate solvent is chosen according to the application method.

4.1.11 Launch the DMS6000 software, and the PLE-EC /GPC.

4.1.12 Open existing or new temperature and pressure log files.

4.1.13 Open the PLE extraction program.

4.1.14 Click on “ALL” to execute the program.

To operate the DMS6000 software refer to the DMS6000 operator’s manual.

4.2 Pressure control

4.2.1 PLE pump: The PLE pump is used to fill columns and pressurize as well as maintain the pressure in the PLE columns.

4.2.1.1 When the PLE is powered up the pump displays the digits 0.0, while the “ML/MIN” yellow LED (flow rate LED) is on. When the pump is running the actual flow rate is displayed.

4.2.1.2 Press the “MODE” button once to display PSI mode. The “PSI” yellow LED (pressure LED) will be lit. In this mode the pump set pressure is displayed.

The set pressure is changed by pressing the “UP ARROW” or “DOWN ARROW”. Press the “UP ARROW” to increase the set pressure or the “DOWN ARROW” to decrease it.

4.2.1.3 Press the “MODE” button once more to display the pump high pressure and the “HIGH PRESSURE” yellow LED will be lit. For the PLE this pressure must always be set at 2600 PSI.

4.2.1.4 Press the “MODE” button once more to display the pump low pressure and the “LOW PRESSURE” yellow LED will be lit. For the PLE this pressure must always be set at 0 PSI.

4.2.1.3 Press the “MODE” button once more to display the actual pressure down stream of the pump. In this mode the “PSI” yellow LED will be blinking.

The PLE pump controls the pressure down stream by its set pressure. When the pressure down stream gets close to the set pressure the pump slows and ultimately stops when the pressure reaches the set pressure.

When for any reason there is a pressure drop down stream and the pressure drops below the set pressure, the pump starts to pump to compensate for the loss of pressure and maintain the pressure at or close to the set pressure.

The PLE pump set pressure must be adjusted to an approximate setting close to the pressure relief valve pressure setting.

4.2.2 The pressure relief valve: The pressure relief valve can be set to open and relieve the pressure in the PLE column and consequently keep the pressure at a constant level.

When heaters turn on and the PLE column is heated the pressure inside will rise. To maintain the pressure inside the PLE column at the pressure specified in the extraction method (P), the pressure relief valve opens to release small amounts of extract to maintain the pressure at P.

The pressure relief valve can be adjusted by turning the cap with the red band around it, located on top of the pressure relief valve.

Turning clockwise will increase the set pressure and counter clockwise will decrease it.

4.2.3 Software pressure control: For a description of this feature please refer to the DMS6000 software operator's manual, section a.2.

4.2.3.1 Every PLE channel has its own pressure transducer (channel pressure transducer). Each channel pressure transducer must be calibrated according to the instructions in the DMS6000 software operator's manual. During the PLE operation each channel pressure transducer sends pressure reading signals for its own channel to the PLE computer through the PLE control module, to be plotted in graphic format on the PLE computer screen.

WARNING: Please make sure to double check the pressure reading on the PLE computer screen and the mechanical pressure gauge to make sure the channel pressure transducers are calibrated and read the correct pressure.

4.2.3.2 The manual valve in each channel is used both to isolate the particular channel from the others and pressure calibration.

For calibrating the channel pressure transducers (pressure calibration) please refer to the DMS6000 software operator's manual.

To isolate a channel from the other channels simply turn the manual valve 90 degrees clockwise.

For example if you have a PLE6 (6 sample PLE system), and you only have 4 samples to run, then you turn the manual valves for channel 5 and 6 , 90 degrees clockwise to isolate channels 5 and 6 and load your samples in channels 1, 2, 3, and 4. Also, you should disable the temperature contro switch for channels 5 and 6 on the PLE control module.

Warning: You must always open temperature and pressure log files for all channels, no more, no less.

For example if you have a PLE3, always open 3 temperature and 3 pressure log files, no more, no less. Even if you are running 2 samples you must open 3 log files for a PLE3, otherwise the readings on the computer screen will be completely incorrect.

4.2.3.3 Mechanical pressure gauge:

The mechanical pressure gauge is used to monitor the pressure by the operator as well as for pressure calibration.

The DMS6000 software operator's manual gives instructions on how to do pressure calibration.

4.3 Temperature control

The PLE computer monitors, displays, and controls the heating of the PLE columns. It receives the temperature probe signals from each channel and according to the temperature set point specified in the program will turn the heater blocks for each channel on or off to maintain the temperature as specified.

To do this accurately the temperature probe for each channel must be calibrated according to the instructions in the DMS6000 operator's manual.

Appendix A: Switches, etc.

1. The Main Power Switch: located on the front side of the PLE control Module.
2. The PLE valve module power switch: Located on the upper right side of the PLE valve module front plate, next to the pressure gauge.

This is the power switch for the individual PLE valve module .

3. PLE control module control switches: located on the front side of the PLE control module.

3.a. Heater control switches:

There are six 3 position toggle switches to control the heaters for channels 1 through 6.

In the down position the heaters are controlled automatically through the software.

In the middle position the heaters are disabled.

In the up position the heaters are controlled manually (They stay on).

3.b. Fans control switch:

There is one 3 position toggle switch to control the fans for channels 1 through 6.

In the down position the fans are controlled automatically through the software.

In the middle position the fans are disabled.

In the up position the fans are controlled manually (They stay on).

3.c. Pump control switch:

There is one 3 position toggle switch to control the pump.

In the down position the pump is controlled automatically through the software.

In the middle position the pump is disabled.

In the up position the pump is controlled manually (it keeps pumping).

Appendix B: Indicator LED'S:

1. Interconnect PC board indicator LED'S:

NOTE: Interconnect PC boards are located in the rear of the large cabinets. They are used to interconnect the PLE valve modules.

The indicator LED'S are red and located on the top of interconnect PC boards. These are voltage indicator LED's. They indicate that the connection for that certain voltage is not broken. (These voltages are necessary for the PLE valve module to function)

1.a L1 indicates **xxxxxv**

1.b L2 indicates **xxxxxv**

1.c L3 indicates **xxxxxv**

2. Fuse, voltage, & control indicator LED'S on the PLE control module:

2.a Voltage indicator LED'S are green and their "ON" state indicate that this particular voltage supply is functioning properly.

2.b Fuse indicator LED'S are yellow and their "ON" indicate that the particular fuse is good.

The fuse indicator LED'S correspond to the silk-screened fuse holders in the rear of the module.

2.c "HTR1" through "HTR6" indicator LED's are red and correspond to the heaters for channels 1 through 6. Their "ON" state indicate that the particular channel heaters are on.

2.d The "pump" indicator LED is red and its "ON" state indicates that the pump is running.

2.e The "FANS" indicator LED is red and its "ON" state indicates that the fans for all channels are blowing.

3. Fuse, voltage, & control indicator LED'S on the PLE valve module:
 - 3.a Voltage indicator LED'S are green and their "ON" state indicate that this particular voltage supply is functioning properly.
 - 3.b Fuse indicator LED'S are yellow and their "ON" indicate that the particular fuse is good.

The fuse indicator LED'S correspond to the silk-screened fuse holders in the rear of the module.
 - 3.c The HTR indicator LED is red and corresponds to the heaters for the particular channel. Its "ON" state indicates that the particular channel heaters are on.
 - 3.d The "FANS" indicator LED is red and its "ON" state indicates that the fans for this particular channel are blowing.

Appendix C: HPLC pump controls & display

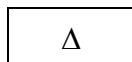
Digital Display:

The 3-digit display shows the pump flow rate (mL/min), system pressure (psi), or the set upper or lower pressure limit (psi) when operating. Choice of display is selected with the MODE key.

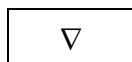
Keypad :



When pressed, this button alternately starts and stops the pump.



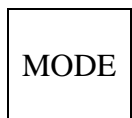
When pressed, this button increases the flow rate.



When pressed, this button decreases the flow rate.



When the PRIME button is pressed, the pump runs at the maximum flow rate for the pump head. It will stop when any button is pressed.



Use this button to cycle through the four display modes: flow rate, pressure, upper pressure limit, or lower pressure limit. A status LED to the right of the digital display indicates which mode is active.

Fast And Slow Button Repeat On The Up And Down Arrow Buttons: If the UP-ARROW or DOWN-ARROW button is held down for more than approximately one half of a second, the button press will repeat at a slow rate of approximately 10 times a second. Once slow button repeat has begun, fast button repeat can be initiated by using a second finger to press down the second arrow button. During fast button repeat, the button press will repeat at a rate of approximately 100 times a second. Switching back and forth between repeat speeds can be accomplished by pressing and releasing the second arrow button while keeping the first arrow button held down.

Status LEDs :

ML/MIN	When lit, the digital display shows flow rate in mL/min.
PSI	When lit, the digital display shows system pressure in psi.
HI PRESS	When lit, the display shows the user-set upper pressure limit in psi.
LO PRESS	When lit, the display shows the user-set lower pressure limit in psi.
PUMP RUN	Lights to indicate that the pump is running.
FAULT	Lights when a fault occurs and stops the pump.

Appendix D: Labels & Markings



symbol indicates: Risk of electric shock



symbol indicates: Hot surface



symbol indicates: caution, risk of danger