

# **Power-Prep™ Cleanup System**

# **User's Manual**

# **FMS**

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## WARNINGS / CAUTIONS

1. The Power-Prep Systems are flexible, yet highly sensitive instruments. Read the User's Manual before use.
2. Only adequately trained personnel should operate the system. A documented training program based on training received during installation from installation engineers is highly recommended
3. Do not shut off the main power switch or individual pump module power switch 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. Do not power the system on after installing module (s) before screwing the module to the cabinet.
8. Always operate the system under specified electrical specifications.
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 acids 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.

## **WARNINGS / CAUTIONS (cont)**

16. To prevent the loss of samples, make sure sufficient solvent exists for the run.
17. Replace waste 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 the tubing. This may restrict flow or cause tubing to fail.

## **DIOXIN SAMPLE CLEAN-UP METHOD USING POWER-PREP**

**Required materials:** The Power-Prep is capable of three sample clean-up runs per day. The following materials are required for three sets of 5 sample runs using the Power-Prep/PP5.

- 1. System, etc.:**
  - 1.a Power-Prep/PP5 System.
  - 1.b Desk top PC, windows 2000 or NT, and the DMS 6000 software rev. 1.09d
  - 1.c RS232 cable connector.
  
- 2. Glassware, and Tubing:**
  - 2.a Fifteen conical glass, sample tubes (od=5/8")
  - 2.b Fifteen sample tubing
  - 2.c Five fraction collector tubing
  - 2.d Fifteen 100-200 mL round bottom glass flasks
  - 2.e Five 125 mL glass bottles
  - 2.f One 4-10 liter glass bottle
  - 2.g Five 25 mL glass test tubes with od=5/8"
  
- 3. Columns and solvents, etc.:**
  - 3.a 15 Silica columns
  - 3.b 15 Alumina columns
  - 3.c 15 carbon columns
  - 3.d 90 Teflon Hex Fittings
  - 3.e 90 Large Teflon Frits
  - 3.f Two 4 liter bottles of Hexane
  - 3.g 1200 mL of 2% DCM/Hexane in a 1.5 or 2 liter bottle
  - 3.h One 4 liter bottle containing 3.3 liters of 50% DCM/Hexane (for 2 sets of samples) and one 2 liter bottle containing 1.7 liters of 50% DCM/Hexane (For the third set of samples).
  - 3.i 700 mL of 50% Ethyl acetate/toluene in a 1 liter bottle
  - 3.j One 4 liter bottle of toluene
  
- 4. Miscellaneous:**
  - 4.a One 500 mL Teflon squirt bottle filled with 50% DCM/Hexane
  - 4.b One 500 mL Teflon squirt bottle filled with Hexane
  - 4.c Thin, powder free nitrile gloves
  - 4.d Laboratory tissues (wipes)
  - 4.e Aluminum foil
  - 4.f Stainless steel round bar 1/4" od x 1FT long

## **IMPORTANT CONSIDERATIONS:**

1. Solvent mixtures must be mixed extremely accurately
2. Solvents must be in thoroughly cleaned glass bottles
3. The following items must be cleaned with 50% DCM/Hexane after every sample clean-up run:
  - 3.a Round bottom glass flasks
  - 3.b Teflon Hex fittings
  - 3.c Sample tubing

**NOTE:** For every set of 5 samples being cleaned-up there are 30 Teflon Hex fittings and 5 sample tubings being used. Having 90 Teflon Hex fittings and 15 sample tubing available enables you to have the entire lot cleaned at the end of the day and have them ready for the next day's 3 runs. These 2 items could be cleaned by sonicated in 50% DCM/Hexane for 10 minutes.

4. The following items are disposable and are used for only one sample clean-up and discarded:
  - 4.a Silica column
  - 4.b Alumina column
  - 4.c Carbon column
  - 4.d Conical glass sample tube
  - 4.e Large Teflon frit
5. The round bottom glass flasks are used to collect fractions. These must be cleaned thoroughly according to the particular method used in the lab to clean sample clean-up glassware.
6. Sample extracts must be ready before the Dioxin.dgf sample Prep Program is started. The sample extract must be concentrated to 200  $\mu$ L and put into the glass sample tube. Hexane is then added to the sample extract to make approximately 12 mL total. However, if the sample extract is already in hexane, no concentration is necessary, provided that the volume is brought to 12 mL. You should label this glass sample tube appropriately for identification and records.
7. Samples 2 through 5 are also labeled the same way

## **How to run a set of 5 Samples:**

1. Place 30 Teflon Hex Fittings on a large enough clean sheet of aluminum foil. (Sonicate in 50% DCM/Hexane before use, as described previously) Fig. 1
2. Place 30 large Teflon frits on a sheet of aluminum foil. (These frits must be sonicated in 50% DCM/Hexane before use, as described previously) Fig. 2
3. Insert one large frit into each Teflon Hex fitting as straight and flat as possible. Using a clean ¼" OD stainless steel round bar, push the frit into the fitting as straight as possible, all the way to the bottom. Fig. 3  
Place the fitting back on the aluminum foil. When all 30 Teflon Hex fittings are prepared, then;
4. Take one silica column out of its vacuum sealed bag and screw one Teflon fitting onto each end.  
Use the torque wrench to tighten both fittings properly. Fig. 4

**NOTE:** Over tightening the fittings onto columns may result in damage to the column and/or fitting threads.

5. Take the silica fitting assembly off the column holder marked "silica" and connect the column in its place with the labeled end of the column on the bottom. Fig. 5  
Snap the column in the 2 plastic clips. Fig. 6  
Repeat steps 4 and 5 for 4 more silica columns for positions 2 through 5. Fig. 7

**NOTE:** The labeled end of silica columns is located on the bottom because the acidic silica part of the column is in that section and the solvents and sample flow from bottom to top.

6. Repeat step 4 and 5 for carbon columns. Fig. 8
7. Take the carbon bypass fitting assembly off the column holder where marked "carbon" and connect the columns in its place, and snap it in the plastic clip. Fig. 9  
Repeat this for 4 more carbon columns for positions 2 through 5. Fig. 10
8. Repeat step 4 and 5 for alumina columns. Fig. 11
9. Take the alumina fitting assembly off the column holder marked "Alumina" and connect the column in its place. Fig. 12  
Snap the column in the 2 plastic clips.  
Repeat this for 4 more alumina columns for positions 2 through 5. Fig. 13
10. Place the 4-10 liter glass bottle or jug under the system inside the cart and insert the waste tubing into it. Fig. 14.

11. Start the Dioxin.dgf program. Refer to the DMS6000 Software operator's manual and appendix A.
12. Watch the connectors on the columns and lines for the first 10 minutes after starting the program closely to ensure they don't leak from any junctions. Fig. 15

**NOTE:** If you notice even a slight leak from the Teflon hex fitting, gently snap the column out of the plastic clips, and tighten the fitting some more, and snap it back in after confirming the leak has stopped. Fig. 16.

13. The glass sample tube #1 is placed in the plastic clip on the sample holder marked "sample" very gently to prevent splashing of the "sample" (position 1 is the first from the left).  
The clean sample tubing (sonicated in 50% DCM/Hexane) is connected to the quick disconnect fitting above the sample holder and the other end of it immersed into the glass sample tube. Fig. 17  
Glass sample tubes #2 through 5 are placed on positions 2 through 5 the same way.

**IMPORTANT NOTE 1:** The sample is loaded onto the silica column in less than 30 minutes of starting the Dioxin.dgf program. Therefore, it is imperative that the glass sample tube containing the sample is placed on the system as soon as possible.

**IMPORTANT NOTE 2:** The Dioxin.dgf program takes approximately 90 minutes to run start to finish. The sample fraction is collected in the last step (step 25) which takes 15 minutes. Therefore, the round bottom glass flasks must be cleaned and ready for use before the fraction collection step (step 25) starts. The round bottom glass flasks are placed in front of their respective positions.

14. The fraction collector tubing for each of the positions is connected to the valve module "Fraction 1" quick disconnect fitting. Insert each fraction collector tubing into its respective round bottom glass flask ensuring the tip of the tubing does not reach the eventual level of fraction collected. Fig. 18
15. When the Dioxin.dgf program ends and the pumps stop, wait until the pressure on the pump module pressure switch decreases to zero before removing the columns from the column holders.



**IMPORTANT NOTE 3:** The Teflon Hex Fittings come with male luer fittings on them, for quick connect/disconnect. To connect the columns to their respective tubing do the following:

- a. Connect the column to the bottom tubing by holding the column with one hand and the female luer part of the tubing with the other, then insert the male luer stem into the female luer part and push together, and turn the female luer clockwise. Fig.19
  - b. Connect the column to the top tubing by holding the column with one hand and the female luer part of the top tubing with the other. Then insert the male luer stem into the female luer port and push together and turn the column clockwise. Fig. 20
  - c. To disconnect and take the columns off, the system repeat steps a and b, except that you will be pulling the fittings away from each other and turning clockwise.
16. After removing the columns, and placing them on a sheet of aluminum foil, reconnect the column fitting assembly.
  17. You can now remove the fractions from the tray and to the evaporator/concentrator for evaporation and eventually GC/MS analysis.
  18. Replace the fraction flasks with the 125 mL. Glass bottles (wash bottles), and drop the fraction collector tubing in their respective bottles.
  19. Discard the conical glass sample tubes.
  20. Replace the contaminated sample intake tubing with clean ones, and put away the contaminated ones to be cleaned at the end of the day.
  21. Place the 25 mL glass test tubes where the conical glass sample tubes were, and insert the clean sample tubing into them.

**NOTE:** These test tubes are “wash vials”

22. Fill the wash vials with 50% DCM/Hexane
23. Run the wash 1 program.
24. When the wash 1 program ends, fill the wash vials with Hexane.
25. Run the wash 2 program.
26. When the wash 2 program ends, the system is ready for the next sample run.

## Appendix A: PC Communication Set-up

1. Turn the main power on  
Note: The main Power Switch is located in the front part of the power supply module, which is located on the top right corner of the system.
2. The control module (located right below the power supply module) will go through a sequence at three initial messages until it settles on the following:  
  
“Multiple process”?
3. Please do the following:  
Control Module Displays
  - “Multiple process?” “NO”
  - “Main Menu?” “NO”
  - “PH Menu?” “NO”
  - “UV Menu?” “NO”
  - “Service Menu?” “YES”
  - “PC Communication?” “YES”
  - “PC Interface” “YES”

The preceding actions set the Power-Prep System Up for PC Communication.

## Appendix B: Switches, etc.

1. The Main Power Switch: located on the front side of the Power Supply Module. Fig. 1
2. The Stand-by switch; located on the front side of the control module (bottom, right). This switch is used to put the system on stand by when a problem occurs (i.e. when the operator forgets to load the sample on the system, etc.) Fig.2

When this switch is turned on; the stand by mode is activated (the system goes on stand by).

When this switch is turned on the “Halt PC” button on the computer monitor must be clicked as well.

3. Valve drive module power switch: located on the front side of the valve drive module.

This is the power switch for the individual valve drive module only. Fig. 3

4. Pump module switches, etc.

- 4.a Pump module power switch:  
Located on the front side of the pump module (lower left side).

This is the power switch for the individual pump module only. Fig 4

- 4.b Pump module pressure reset switch:  
Located on the front side of the pump module, on the left hand side, and below the red LED. Fig. 5

This switch is used to reset the system after an overpressure problem has been resolved, press the reset switch to reset the system.

- 4.c Pump module motor enables toggle switch:  
Located on the front side of the pump module, on the left hand side and below the reset switch. Fig. 6

This switch is used to enable/disable the individual pump module.

When the switch is toggle down the pump module is enabled, and when it is in the middle or up state the pump module is disabled.

- 4.d Pump module pressure switch:  
Located on the front side of the pump module and above the pump-head. Fig. 7

**The Pressure Switch has two functions:**

1. It shows the pressure on the outlet of the pump-head at any given moment during system operation.
2. It places the system on stand by if an overpressure situation occurs.

The pressure switch has a range of 0-30 PSI.

Using the set pressure screw the maximum set pressure can be set by turning the screw CCW the set pressure will increase and CW it will decrease.

For Example: If the maximum set pressure is set at 25 PSI, then the system will be put on stand by if the pump outlet pressure exceeds 25 PSI. Fig 8

**NOTE:** FMS Systems are shipped with the maximum set pressure set at 30 PSI.

4.e Pump-head:

The pump-heads used in the Power-Prep Systems are positive displacement piston pump-heads. The wetted parts are PTFE Teflon, stainless steel and carbon. The pump-head fluid displacement is in direct proportion to the stroke length.

The stroke length is increased by turning the black knob on the front left hand side of the pump-head. Fig. 10

The “Front pointer” of the pump-head point to a specific number on the “stroke length range” Fig. 11

The “Stroke length range” is divided into two mirror sections (numeral “O” in the middle and incrementing from 1 through 10 in opposite directions).

By adjusting the “front pointer” to the left of “O” the outlet port is on the left and inlet port on the right, and to the right of “O” the reverse. Fig. 12.

## **Appendix C: Indicator LED'S:**

1. Interconnect PC board indicator LED'S:

**NOTE:** Interconnect PC boards are located in the rear of the different cabinets. They are used to interconnect the various modules.

The indicator LED'S are red and located on the top right corner of interconnect PC boards.

The indicator LED'S are convenient troubleshooting tools, in that they indicate the required electrical voltage for a particular module, while a specific LED is on, the corresponding voltage (which is silk-screened beside the LED) is present on the same interconnect PC board.

2. Fuse and voltage indicator LED'S:

- 2.a Voltage indicator LED'S are green and indicate the presence of the required voltages inside the particular module. (The LED being "ON" indicates the presence of the specific voltage).

- 2.b Fuse indicator LED'S are yellow and when "on" indicate that the particular fuse is good at present.

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

## Appendix D: Numbers

1. Solvent volumes per sample clean-up run:

Hexane=324 mL  
2% DCM / Hexane=72mL  
50%DCM / Hexane=164mL  
50%Ethyl Acetate / Toluene=38mL  
Toluene=139mL

2. Solvent volumes for 2 wash program runs:

Hexane=190mL  
50%DCM / Hexane=190mL

3. Approximate pure solvent volumes per sample:

Hexane=770mL  
DCM=170mL  
Toluene=155mL  
Ethyl Acetate=20mL

## Appendix E: Pump Selection Calibration

To set the pump option and calibrate the pump do the following:

### 1-Select Option Menu:

<u>LCD Displays</u>	<u>Operator Press on Keypad</u>
Multiple Process	“No”
Main Menu?	“No”
PH Menu?	“No”
UV Menu?	“No”
Service Menu?	“No”
Option Menu?	

### 2- Select settings:

<u>LCD Displays</u>	<u>Operator Press on Keypad</u>
Option Menu?	“Yes”
Pump Option?	“Yes”
Peristaltic Pump Head?	“Yes”
Buzzer Option?	“yes”
Select Buzzer? Yes      No	“yes”
Valve Option?	“yes”
Select valves? Yes      No	“yes”

How many valve Modules?<?>	“8”,then “enter”
Set time	“yes”
Sample 1?	“yes”
Enter 0000.00	“0.04”, “enter”
Volume Option?	“yes”
Volume in ml?	“yes”
Service Menu?	“No”
Option menu?	“No”
Main menu?	

### 3- Calibrate Pump:

#### LCD Displays

#### Operator Press on Keypad

Main Menu?	“Yes”
Calibration?	“Yes”
ID=13? Flow: -02-15 ML/M	“Yes”
Select One of 6 M1: Valve <0>	“0” Then “Enter”
Select One of 6 M2: Valve <0>	“1” Then “Enter”
Select One of 6	



M3: Valve <0>	“2” Then “Enter”
Select One of 6 M4: Valve <0>	“2” Then “Enter”
Select One of 6 M5: Valve <0>	“2” Then “Enter”
Select One of 6 M6: Valve <0>	“0” Then “Enter”
Select One of 6 M7: Valve <0>	“0” Then “Enter”
Select One of 6 M8: Valve <0>	“6” Then “Enter”
How many Pump Heads <1>	“1” Then “Enter”
Input Flow rate To be calibrated	“Yes”
Flow? 0000-00	“10” Then “Enter”
Please Wait	
Ready to measure Volume	“Yes”
Calibration Dispensing....	

**Note: The pump runs for approximately 1 minute and then will prompt the following:**

**LCD Displays**

**Operator Press on Keypad**

Input volume Dispensed	“Yes”
Volume= 0000-00	“10” Then “Enter”

Calibration Complete	“yes”
Multiple Process	“No”
Main Menu?	“No”
PH Menu?	“No”
UV Menu?	“No”
Service Menu?	“Yes”
PC-Communication	“Yes”