

Suggested Procedure for Using Empore™ 47 and 90 mm Oil & Grease Disk Extraction Disks with the Horizon SPE-DEX 4790 Extractor and SPE-DEX Controller

The program below has been validated within 3M using two lots of 47 mm Empore™ Oil and Grease Extraction Disks for the recovery of the hexadecane/stearic acid standard specified in EPA Method 1664 Revision A. Adjustments to the protocol may be needed to accommodate specific requirements.

Results obtained within the 3M laboratory are as follows:

	Lot 1	Lot 2
% Recovery (n=7)	94 %	94 %
% Std. dev.	2.340126	4.220133

Tips for Optimizing Results

1. Run a blank.
2. The Empore™ Oil & Grease Disks can be soaked overnight (prior to use) in hexane or acetone to minimize blank contributions.
3. A purge protocol may be needed between extractions of dirty samples to ensure the extractor system is clean.

Programming the Horizon SPE-DEX Controller for EPA Method 1664A

1. Press the method key on the controller.
2. Enter a number to identify the new method.
3. Record the new method number and test method for future reference.
4. Enter the information from the table below into the controller.

Prewet Step 1		Prewet Solvent 1
Soak	1:00	Hexane
Air dry	1:30	
Prewet Step 2		Prewet Solvent 2
Soak	1:00	Methanol
Air dry	0:00	
Air dry step for filtered disk		
Air dry	5:00	
Rinse Step 1		Rinse Solvent 1
Soak	0:30	Hexane
Air dry	0:30	

Rinse Step 2		Rinse Solvent 1
Soak	0:00	Hexane
Air dry	0:02	
Rinse Step 3		Rinse Solvent 1
Soak	0:00	Hexane
Air dry	0:02	
Rinse Step 4		Rinse Solvent 1
Soak	2:00	Hexane 1
Air dry	2:00	
Rinse Step 5		Rinse Solvent 1
Soak	1:00	Hexane
Air dry	1:00	
Rinse Step 6		Rinse Solvent 1
Soak	2:00	Hexane
Air dry	2:00	

Add the purge program (if needed):

1. Press the method key on the controller.
2. Enter a number to identify the new purge method.
3. Record the new purge method number and purge method for future reference.
4. Enter the information in the table below into the controller*.

Prewet Step 1		Prewet Solvent 1
Soak	0:00	Hexane
Air dry	5 seconds	
Prewet Step 2		Prewet Solvent 2
Soak	0:00	Methanol
Air dry	10 seconds	

**Adjustments to the protocol may be needed to accommodate specific samples or requirements.*

Set Up Procedure

1. Check that solvent bottles are filled. Ensure the solvent lines are not kinked or crimped.
2. Empty both waste bottles (water and solvent)
3. Turn on the vacuum pump source (20 - 25 in. Hg is recommended)
4. Turn on the gas supply (40 - 60 psi)
5. Check SPE Extractor pressure (30 - 50 psi)
6. Check solvent bottle gas pressure (10 -15 psi)
7. Check solvent waste vacuum setting (10 -15 inches Hg)
8. Use the check valve tool to gently free check valve.
9. Load an empty disk holder cup with screen onto platform.
10. Switch the controller on. (The switch is located on the back of controller)
11. Attach a collection vial.
12. Place empty sample bottle in extractor.
13. Press select key on the controller.
14. Enter the purge method number to be used and number of extractor(s).
15. Press purge on the extractor. (Should take approximately 2 minutes to run).
When the red standby light goes on the cycle is complete.
16. Remove collection vial, empty and replace.
17. Purge a second time. (Eliminates air in lines and rinses lines from previous sample).
18. Remove sample bottle and collection vial.
19. Place water sample bottle and clean collection vial/flask in extractor.
(11dram vial with collection vessel 19/22 adapter or 50 ml flask 19/22 neck for 47 mm disk and 250 ml flask 19/22 neck for 90mm disk)
20. Place disk in the center of the gasket over the screen, dimpled side down, into the holder. The collar should be place with the thin groove side down on the gasket. Next, screw the aluminum ring onto the disk holder base. Check to ensure the disk is tight.
21. Press select key on the controller.
22. Enter the method number and the number of extractor(s) into the controller.
23. Press the Start/Run key on the extractor. The test should take approximately 45 minutes to run. When the red standby light goes on the cycle is complete.
24. Remove the eluate sample vial/flask and process according to EPA method 1664A.

Shut Down Procedure

1. Remove the sample bottle. Cover the opening to prevent airborne debris from depositing in the holder. Aluminum foil, Saran Wrap™ or other materials may be used.
2. Fill the Empty Disk Holder about half full with water.
3. Attach a vial to collect the purge rinse.
4. Hold down Fault/Abort key and press PURGE key. (This will flush and clean the eluate check valve of residual sample or debris)
5. Release Fault/Abort key when Start/Run light goes on.
6. Turn off vacuum pump and vent by disconnecting the line to the waste water bottle for 5 minutes.
7. Turn off gas supply.
8. Turn off power supply.