EPA Method 1664A–Extraction of Oil and Grease from Water Samples Using the Thermo Scientific Dionex AutoTrace 280 Solid-Phase Extraction Disk Configuration

INTRODUCTION

EPA Method 1664, Revision A, *n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel-Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry*, describes the determination of oil and grease from liquid (typically water) samples. Liquid-liquid extraction (LLE) and solid-phase extraction (SPE) are both proven techniques that meet the requirements of EPA 1664A. Although generally effective, this method may be very time-consuming, labor-intensive, and use large amounts of solvent. SPE is a good alternative to liquid-liquid extraction. The SPE technique provides a more reliable, less labor-intensive solution to liquid extraction, and uses far less solvent than LLE. Also, emulsion formation is eliminated by using SPE. The Thermo Scientific Dionex AutoTrace™ 280 SPE instrument automates conditioning, rinsing, loading, drying, and extraction and also can simultaneously process 1 to 6 samples. The AutoTrace 280 SPE instrument is available in both disk and cartridge configurations to suit the needs of every laboratory.

This application note describes the use of the Dionex AutoTrace 280 instrument to extract oil and grease from water samples in accordance with the EPA Method 1664, Revision A.

EQUIPMENT

Dionex AutoTrace 280 Automated Large Volume SPE for Disks (P/N 071386)

pH paper, range 1–2.5, (Fisher Scientific)

SOLVENTS

Hexane, pesticide-grade or equivalent (Fisher Scientific)
Methanol, pesticide-grade or equivalent (Fisher Scientific)
Water, HPLC-grade or equivalent (Fisher Scientific)
HCL, 12 M

SPE MATERIAL

Empore™ High Performance Extraction Disks, (P/N 2215, 3M)

OIL AND GREASE DISC CONFIGURATION METHOD

Acidify each 1 L sample to pH < 2 using 6 M HCl. Place required number of samples (1–6) in the sample vial rack. Insert sample lines into each sample bottle.

Collection

Label the collection vials (1–6) and place these into the collection rack. Position the solvent bottles on the left side of the Dionex AutoTrace system.

Solvents

Add hexane to solvent bottle 1, methanol to solvent bottle 2, and HPLC water (pH = 2) to solvent bottle 3. Insert the solvent lines into the corresponding bottle. (If desired, these lines can also be labeled 1–3. Up to five different solvents can be used with the AutoTrace system).

SPE Media

Insert SPE disks onto the Dionex AutoTrace system (see *Dionex AutoTrace 280 Operation Manual* for details[1]) and secure the disk into place using the disk holder. The green LED will be illuminated when the disk is locking into place.

Solid-Phase Extract

No.	Method	User Intervention
1	Process 6 samples using the following method steps	
2	Wash syringe with 10.0 of hexane	
3	Condition disk with 10.0 mL of hexane into solvent waste	
4	Condition disk with 10.0 mL of hexane into solvent waste	
5	Dry disk with gas for 5.0 min	
6	Wash syringe with 10.0 mL of CH_3OH	
7	Condition disk with 10.0 mL of CH_3OH into solvent waste	
8	Wash syringe with 10.0 of water (pH = 2)	
9	Condition disk with 10.0 mL of water into aqueous waste	
10	Load 1050.0 mL of sample onto disk	
11	Pause and Alert operator, resume when CONTinue is pressed	Rinse sample bottles with 50 mL water
12	Load 100.0 mL of sample onto disk	
13	Dry disk with gas for 10.0 min	

Solid-Phase Elute

No.	Method	User Intervention
14	Manually rinse sample container with 20.0 mL to collect	Rinse with 10 mL hexane. Remove collecion vials and keep. This is fraction 1. Replace with new collection vials.
15	Manually rinse sample container with 20.0 mL to collect	Rinse with 10 mL hexane. Remove collecion vials and keep. This is fraction 2. Replace with new collection vials.
16	Pause and Alert operator, resume when CONTinue is pressed	
17	Wash syringe with 10.0 mL of CH_3OH	
18	Wash syringe with 10.0 mL of hexane	
19	Soak and collect 3.0 mL fraction using hexane	
20	Pause for 2.0 min	
21	Collect 2.0 mL fraction into sample tube using hexane	
22	Soak and collect 3.0 mL fraction using hexane	
23	Pause for 2.0 min	
24	Collect 2.0 mL fraction into sample tube using hexane	Rinse with 10 mL hexane. This is fraction 3. Combine fractions 1, 2, and 3. Evaporate solvent for gravimetric determination.
25	Concentrate sample with gas for 1.0 min	
26	End	

Standard Solutions

Standard stock solution: 400 mg each of hexadecane and stearic acid into 100 mL acetone.

Standard acidification solution: equal parts concentrated HCl (12 M) and water to obtain 6 M HCl.

Sample Preparation

Dispense 1 L of water from the water system.
Add 5 mL of the standard stock solution and mix gently.
Add 10 mL of CH_3OH and mix gently.
Add 3 mL of 6 M HCl and mix gently.
Check pH using pH paper (range 1–2.5) to verify sample pH ≤ 2.

Save and download this program on the AutoTrace system for future use. Note: Up to 24 different programs can be saved onto the AutoTrace system. Methods are written and transferred to the AutoTrace system using a computer. Once the desired methods are stored on the AutoTrace system, there is no further need for the AutoTrace instrument to be connected to the computer.

Enter the following parameters for the extraction method under the Params Tab of the SET UP METHODS screen:

Flow Rates		SPE Parameters	
Cond Flow	40 mL/min	Push Delay	5 s
Load Flow	30 mL/min	Air Factor	1.0
Rinse Flow	40 mL/min	Autowash Vol.	1.00 mL
Elute Flow	5 mL/min		
Cond Air Push	40 mL/min		
Rinse Air Push	40 mL/min		
Elute Air Push	5 mL/min		

Estimated time: 1 h 27 min for 6 samples
Estimated solvent used: 25 mL

Gravimetric Analysis

The elutes and rinses were combined into pre-tared vials, the solvent was evaporated under a gentle stream of nitrogen gas, and the vials were weighed again. The results are presented in Table 1. (Refer to EPA Method 1664, Revision A, section 11.3.12 for instructions to distinguish between n-hexane extractable material and silica gel-treated n-hexane extractable material.)

RESULTS

Table 1 shows the results from an oil and grease single stock solution split into six samples and extracted using the Dionex AutoTrace 280 instrument.

Table 1. Oil and Grease Recoveries From a Stock Solution

Replicate	Sample Weight (mg)	Recovery %*
Disk 1	39.9	99.5
Disk 2	39.3	98.0
Disk 3	39.2	97.7
Disk 4	39.5	98.6
Disk 5	40.0	99.7
Disk 6	38.3	95.5
	Mean	98.2
	St. Dev.	1.5
	% RSD	1.6

** % Recovery = (weight of the recovered hexadecane/steric acid divided by the known weight of the stock solution) x 100*

CONCLUSIONS

The Dionex AutoTrace SPE instrument provides an automated, solid-phase extraction solution to EPA 1664, Revision A that gives excellent recovery and precision and replaces tedious and labor-intensive liquid-liquid extraction.

REFERENCES

1. Thermo Fisher Scientific Inc. *AutoTrace 280 Operator's Manual*, Doc. 065330-01, Sunnyvale, CA.

LIST OF MANUFACTURERS

Thermo Fisher Scientific Inc., 1228 Titan Way, P.O. Box 3603, Sunnyvale, CA 94088-3603, http://www.dionex.com

Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275-1126, Tel: 800-766-7000, www.fishersci.com

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