

EPA Method 1664A—Extraction of Oil and Grease from Water Samples Using AutoTrace 280 Solid-Phase Extraction Cartridge 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, LLE is a very time-consuming, labor-intensive technique that uses 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 Dionex AutoTrace® 280 SPE instrument automates the conditioning, rinsing, loading, drying, and extraction steps 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 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 a 6 mL cartridge (P/N 071385)
pH paper

SOLVENTS

Hexane, pesticide-grade or equivalent (Fisher Scientific)
Methanol (CH₃OH), pesticide-grade or equivalent (Fisher Scientific)
Water acidified to pH 2, 18 MΩ Milli-Q® water system (Millipore)
Tetrahydrofuran (THF), pesticide-grade or equivalent (Fisher Scientific)
Water, 18 MΩ, Milli-Q water system (Millipore)
Hydrochloric acid (HCL)

SPE MATERIAL

Dionex SolEx (P/N 074410), 6 mL, C18 SolEx cartridge with 1.0 g of packing

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OIL AND GREASE CARTRIDGE CONFIGURATION METHOD

Place required number of samples (1–6) in the sample vial rack. Allow all samples to reach room temperature and adjust the pH of each sample to 2 using 6 M HCL. Insert sample lines into each sample bottle.

Collection

Label the required number of collection vials (1–6) and place these into the collection rack.

Solvents

Add methanol to solvent bottle 1, water (pH 2) to solvent bottle 2, hexane/THF (1:1) to solvent bottle 3, hexane to solvent bottle 4, and water to solvent bottle 5. Place these solvent bottles to the left side of the AutoTrace system and insert the solvent lines into the corresponding bottle (up to five different solvents can be used with the AutoTrace system).

SPE Media

Insert SPE cartridges onto the AutoTrace system (see *Dionex AutoTrace 280 Operation Manual* for details¹) and secure the cartridge into place using the cartridge holder. The green LED will be illuminated when the cartridge is locked into place.

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 (six samples)

Dispense 1 l of water from the Milli-Q water system.

Add 5 mL of the standard stock solution and mix gently.

Add 10 mL of CH₃OH and mix gently.

Add 3 mL of 6 M HCl and mix gently.

Program, download, save, and run the following method to the AutoTrace 280 SPE Instrument:

No.	Method	User Intervention
1	Process 6 samples using the following method steps	
2	Wash syringe with 3.0 mL CH ₃ OH	
3	Condition cartridge with 5.0 mL hexane into solvent waste	
4	Condition cartridge with 5.0 mL hexane into solvent waste	
5	Dry cartridge with gas for 5.0 min	
6	Condition cartridge with 5.0 mL CH ₃ OH into solvent waste	
7	Condition cartridge with 5.0 mL water, pH 2 into aqueous waste	
8	Load 1200.0 mL of sample onto cartridge	
9	Dry cartridge with gas for 60.0 min	
10	Manually rinse sample container with 30.0 mL to collect	Rinse sample bottles with 10 mL hexane
11	Pause and alert operator, resume when CONTInue is pressed	Fraction#1. Place new vials in collection rack
12	Concentrate sample with gas for 10.0 min	
13	Wash syringe with 3.0 mL CH ₃ OH	
14	Soak and collect 4.0 mL fraction using hexane THF (1:1)	
15	Pause for 2.0 min	
16	Collect 3.0 mL fraction into sample tube using hexane/THF (1:1)	
17	Soak and collect 3.0 mL fraction using hexane/THF (1:1)	
18	Pause for 2.0 min	
19	Collect 3.0 mL fraction into sample tube using hexane/THF (1:1)	Fraction #2. Combine fractions 1 and 2. Evaporate solvent for gravimetric determination.
20	End	

Enter the following parameters for the extraction method under the Params Tab:

Flow Rates		SPE Parameters	
Cond Flow	10 mL/min	Push Delay	5.0 s
Load Flow	10 mL/min	Air Factor	1.0
Rinse Flow	10 mL/min	Autowash Vol.	2.00 mL
Elute Flow	5 mL/min		
Cond Air Push	10 mL/min		
Rinse Air Push	10 mL/min		
Elute Air Push	5 mL/min		

Estimated time: 4 h 12 min (for 6 samples)

Estimated solvent used: 25 mL

Save and download this program on the AutoTrace system for future use. Note: Up to 24 different programs can be stored on the AutoTrace system. These methods can be 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 to be connected to the computer.

RESULTS

Table 1 shows oil and grease recoveries from a single stock solution split into six samples and extracted using the AutoTrace 280 SPE instrument.

Replicate	Sample Weight (mg)*	Recovery %**
Cartridge 1	36.0	90.0
Cartridge 2	37.5	93.8
Cartridge 3	37.7	94.3
Cartridge 4	36.8	92.0
Cartridge 5	36.9	92.3
Cartridge 6	36.6	91.5
	Mean	92.3
	St. Dev.	1.5
	%RSD	1.7

* The sample weight is a combined weight of both fractions.

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

CONCLUSIONS

The AutoTrace 280 SPE instrument with the cartridge configuration provides an automated, solid-phase extraction solution that replaces traditional liquid-liquid extraction and meets the requirements of EPA 1664, Revision A.

REFERENCES

1. Dionex Corporation, *AutoTrace 280 Operator's Manual*, Doc. 065330-01, Sunnyvale, CA.

LIST OF MANUFACTURERS

Dionex Corporation, 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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Dionex Corporation

1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

North America

U.S./Canada (847) 295-7500

South America

Brazil (55) 11 3731 5140

Europe

Austria (43) 1 616 51 25 Benelux (31) 20 683 9768 (32) 3 353 4294
Denmark (45) 36 36 90 90 France (33) 1 39 30 01 10 Germany (49) 6126 991 0
Ireland (353) 1 644 0064 Italy (39) 02 51 62 1267 Sweden (46) 8 473 3380
Switzerland (41) 62 205 9966 United Kingdom (44) 1276 691722

Asia Pacific

Australia (61) 2 9420 5233 China (852) 2428 3282 India (91) 22 2764 2735
Japan (81) 6 6885 1213 Korea (82) 2 2653 2580 Singapore (65) 6289 1190
Taiwan (886) 2 8751 6655

www.dionex.com



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