

Application

Note
59

Extraction of Organic Compounds from Water, Using ENVI™-18 DSK Solid Phase Extraction Disks

ENVI-Disk solid phase extraction disks are a porous glass fiber matrix containing C8- or C18-modified silica. In extractions of organic contaminants from 1 liter or more of water, these rigid disks provide faster flow rates and exhibit less clogging than Teflon® disks, and are less expensive. They can be used to extract polynuclear (polycyclic) aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, herbicides, and phthalates from water. This application note describes a process for extracting a wide range of organics from water according to US Environmental Protection Agency Method 525.1.

Key Words:

- semivolatiles • water analyses • SPE disks
- sample preparation

NOTE: This method is based on the sample extraction procedure in Section 11.2 of US Environmental Protection Agency Method 525.1. It is not intended to be a replacement or substitute for the EPA procedure. For detailed information about preparing samples for analysis according to Method 525.1, please refer to the EPA method. (Request from National Technical Information Service, U.S. Department of Commerce, Springfield, VA 22161 USA [Tel. 703-487-4650])

Sample Preparation

1. Allow 1 liter of drinking water to equilibrate to room temperature in a narrow mouth amber glass bottle.
2. Adjust sample pH to less than 2 with 6N hydrochloric acid.
3. Add 5mL methanol and mix thoroughly.
4. Add internal standards. For QC/QA samples, add 2µg each analyte (8µg pentachlorophenol) and 5µg each internal standard.

Apparatus Assembly

1. Assemble the 47mm glassware (flask, vacuum line, and filtration support) according to the instructions that accompanied the ENVI-Disk™ SPE disks.
2. Place a 47mm ENVI-18 DSK disk (Cat. No. 57171) on the support, **wrinkled side up**.
3. Center the disk, so that it evenly overlaps all sides of the support.
4. Carefully place the reservoir on the disk and attach the clamp securely.

Disk Cleaning

1. Pour or pipette 5mL methylene chloride onto the disk and immediately draw the liquid through the disk under moderate vacuum (15" Hg/50kPa). Maintain vacuum for 5 minutes, to remove all solvent.

Disk Conditioning

1. Pour or pipette 5mL methanol onto the disk and immediately apply low vacuum (1–2" Hg/3–7kPa). Release the vacuum when the methanol is just above the top surface of the disk. **Do not allow any air to pass through the disk or to reach the top surface of the disk.**
2. Immediately pour or pipette 5mL deionized water onto the disk and immediately apply low vacuum. Release the vacuum when the water is just above the top surface of the disk.
Note: In steps 1 and 2 it is better to leave extra liquid covering the disk than to allow air to contact the surface of the disk.

Sample Addition

1. Pour the sample into the apparatus, directly onto the film of water left on the disk from the last conditioning step. Invert the sample bottle in the reservoir and allow the sample to automatically feed onto the disk.
2. Adjust the vacuum to approximately 10" Hg (35kPa), to provide a flow rate of approximately 100mL/minute. The disk must not go dry until the entire sample has been processed.

Disk Drying

1. After the entire sample has been drawn through the disk, draw air through the disk under moderate vacuum (approximately 15" Hg/50kPa) for approximately 5 minutes.

Analyte Elution

1. Release the vacuum.
2. Remove the filtration support and reservoir from the vacuum flask without disturbing the disk.
3. Empty the processed water from the flask, insert the sample collection tube, and reassemble the apparatus.
4. Add 5mL methylene chloride to the sample bottle, cap the bottle, and gently swirl it to rinse all of the inside surface. Allow the bottle to stand for 1–2 minutes.
5. Transfer the methylene chloride to the disk, using a glass pipette and rinsing the sides of the reservoir in the process.
6. Draw the solvent through the disk (5" Hg/17kPa vacuum).
7. Repeat steps 4–6 two more times with fresh aliquots of methylene chloride, combining all eluates in the sample collection tube.

Analysis

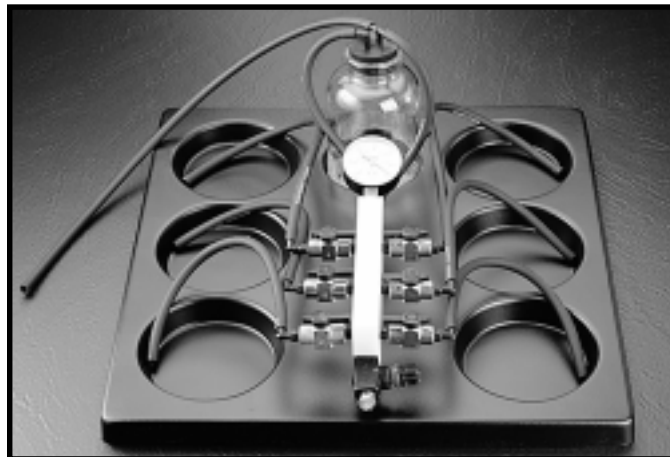
1. Remove any remaining water from the eluate by passing it through approximately 3g anhydrous sodium sulfate.
2. Concentrate the eluate to 1mL and analyze 1µL by GC/MS, following the procedure in EPA Method 525.1.

ENVI-Disk



994-0256

ENVI-Disk Holder Manifold



994-520

ENVI-Disk Holder



995-0117

ENVI-Disk Clamp



996-0280

Reference

1. US Environmental Protection Agency Method 525.1.
Request from National Technical Information Service, U.S. Department of Commerce, Springfield, VA 22161 USA (Tel. 703-487-4650).

Trademarks

ENVI, ENVI-Disk, PTE – Sigma-Aldrich Co.
Teflon – E.I. du Pont de Nemours & Co., Inc.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

Contact our Technical Service Department
(phone 800-359-3041 or 814-359-3041, FAX 814-359-5468)
for expert answers to your questions.

Ordering Information:

Description	Cat. No.
ENVI-18 DSK SPE Disks – C18 bonded phase	
47mm, pk. of 24	57171
90mm, pk. of 12	57170-U
Accessories	
ENVI-Disk Holder*	57173
Flask, 1-liter, 40/35 fitting	Z290610-1EA
Collection Tube, 25 x 250mm	57175
ENVI-Disk Holder Manifold	57174
47mm Filter Clamp Assembly	57260-U
Replacement Teflon Stage, 47mm	57261

* Flask and tube not included with holder.

Note S9

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

ARGENTINA • Sigma-Aldrich de Argentina, S.A. • Buenos Aires 1119 AUSTRALIA • Sigma-Aldrich Pty. Ltd. • Castle Hill NSW 2154 AUSTRIA • Sigma-Aldrich Handels GmbH • A-1110 Wien
BELGIUM • Sigma-Aldrich N.V./S.A. • B-2880 Bornem BRAZIL • Sigma-Aldrich Quimica Brasil Ltda. • 01239-010 São Paulo, SP CANADA • Sigma-Aldrich Canada, Ltd. • 2149 Winston Park Dr., Oakville, ON L6H 6J8
CZECH REPUBLIC • Sigma-Aldrich s.r.o. • 186 00 Praha 8 DENMARK • Sigma-Aldrich Denmark A/S • DK-2665 Vallensbaek Strand FINLAND • Sigma-Aldrich Finland/YA-Kemia Oy • FIN-00700 Helsinki
FRANCE • Sigma-Aldrich Chimie • 38297 Saint-Quentin-Fallavier Cedex GERMANY • Sigma-Aldrich Chemie GmbH • D-82041 Deisenhofen GREECE • Sigma-Aldrich (o.m.) Ltd. • Ilioupoli 16346, Athens
HUNGARY • Sigma-Aldrich Kft. • H-1067 Budapest INDIA • Sigma-Aldrich Co. • Bangalore 560 048 IRELAND • Sigma-Aldrich Ireland Ltd. • Dublin 24 ISRAEL • Sigma Israel Chemicals Ltd. • Rehovot 76100
ITALY • Sigma-Aldrich s.r.l. • 20151 Milano JAPAN • Sigma-Aldrich Japan K.K. • Chuo-ku, Tokyo 103 KOREA • Sigma-Aldrich Korea • Seoul MALAYSIA • Sigma-Aldrich (M) Sdn. Bhd. • Selangor
MEXICO • Sigma-Aldrich Química S.A. de C.V. • 50200 Toluca NETHERLANDS • Sigma-Aldrich Chemie BV • 3330 AA Zwijndrecht NORWAY • Sigma-Aldrich Norway • Torshov • N-0401 Oslo
POLAND • Sigma-Aldrich Sp. z o.o. • 61-663 Poznań PORTUGAL • Sigma-Aldrich Quimica, S.A. • Sintra 2710 RUSSIA • Sigma-Aldrich Russia • Moscow 103062 SINGAPORE • Sigma-Aldrich Pte. Ltd.
SOUTH AFRICA • Sigma-Aldrich (pty) Ltd. • Jet Park 1459 SPAIN • Sigma-Aldrich Quimica, S.A. • 28100 Alcobendas, Madrid SWEDEN • Sigma-Aldrich Sweden AB • 135 70 Stockholm
SWITZERLAND • Supelco • CH-9471 Buchs UNITED KINGDOM • Sigma-Aldrich Company Ltd. • Poole, Dorset BH12 4QH
UNITED STATES • Supelco • Supelco Park • Bellefonte, PA 16823-0048 • Phone 800-247-6628 or 814-359-3441 • Fax 800-447-3044 or 814-359-3044 • email: supelco@sial.com

H

Supelco is a member of the Sigma-Aldrich family. Supelco products are sold through Sigma-Aldrich, Inc. Sigma-Aldrich warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product for a particular use. Additional terms and conditions may apply. Please see the reverse side of the invoice or packing slip.

AGU