

Extraction of Paraquat and Diquat from Water, Using ENVI™-8 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 paraquat and diquat from water according to US Environmental Protection Agency Method 549.1.

Key Words:

- paraquat ● diquat ● pesticides ● water analyses
- SPE disks ● sample preparation

NOTE: This method is based on the sample extraction procedure in Section 11.3 of US Environmental Protection Agency Method 549.1. 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 549.1, 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])

Solution Preparation

Conditioning Solution A: Dissolve 0.500g cetyl trimethyl ammonium bromide and 5mL concentrated ammonium hydroxide in 500mL deionized water. Dilute to 1000mL in a volumetric flask.

Conditioning Solution B: Dissolve 10.0g 1-hexanesulfonic acid (sodium salt) and 10mL concentrated ammonium hydroxide in 250mL deionized water. Dilute to 500mL in a volumetric flask.

10% (w/v) Sodium Hydroxide: Dissolve 50g sodium hydroxide in 400mL deionized water. Dilute to 500mL in a volumetric flask.

10% (v/v) Hydrochloric Acid: Add 50mL concentrated hydrochloric acid to 400mL deionized water. Dilute to 500mL in a volumetric flask.

Disk Eluting Solution: Add 13.5mL orthophosphoric acid and 10.3mL diethylamine to 500mL deionized water. Dilute to 1000mL in a volumetric flask.

Ion Pair Concentrate: Dissolve 3.75g 1-hexanesulfonic acid in 15mL disk eluting solution. Dilute to 25mL with disk eluting solution in a volumetric flask.

Sample Preparation

1. Allow a 250mL sample of water to equilibrate to room temperature in a silanized amber glass bottle or an amber polyvinylchloride bottle.

2. Adjust sample pH to 10.5 ± 0.2 with 10% (w/v) sodium hydroxide or 10% (v/v) hydrochloric acid.
3. Extract sample immediately after adjusting pH.
4. For QC/QA samples, add 25µg of each analyte.

Apparatus Assembly

1. Assemble the 47mm glassware (flask, vacuum line, and filtration support) according to the instructions that accompanied the ENVI-Disk™ SPE disks. Silanization of the apparatus might improve analyte recovery.
2. Place a 47mm ENVI-8 DSK disk (Cat. No. 57172) 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 Conditioning

Pour or pipette the following solvents onto the disk in the sequence indicated.

Apply low vacuum (1–2" Hg/3–7kPa) immediately after adding the first solvent (methanol), and maintain the vacuum throughout the conditioning sequence.

When the level of one solvent is just above the top surface of the disk add the next solvent.

Do not allow any air to pass through the disk or to reach the top surface of the disk.

Solvents:

1. 10mL methanol
2. 2 x 10mL deionized water
3. 10mL conditioning solution A
4. 2 x 10mL deionized water
5. 10mL conditioning solution B

Sample Addition

1. Pour the sample into the apparatus, directly onto the film of conditioning solution B left on the disk from the last conditioning step.
2. Increase 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.

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 a silanized glass sample collection tube, and reassemble the apparatus.
4. Add approximately 1 mL methanol to the disk. Be sure to cover the entire surface of the disk.
5. Immediately add 4 mL disk eluting solution and draw the solution through the disk at 1–2" Hg (3–7 kPa) until the solution just covers the top surface of the disk.
6. Add an additional 4 mL of disk eluting solution and draw it completely through the disk.

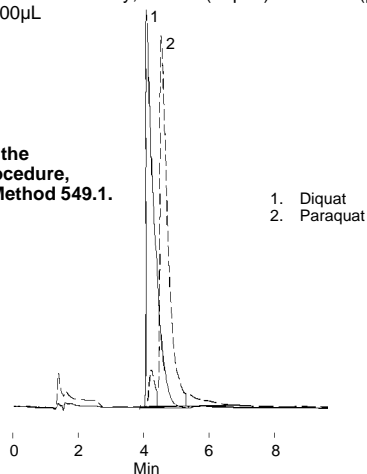
Analysis

1. Add 100 µL ion pair concentrate to the eluate.
2. Adjust volume as necessary with elution solvent and analyze 100 µL by HPLC/UV.

Figure A. Paraquat and Diquat Extracted from Water, Using an ENVI-8 DSK SPE Disk

Sample: 250 mL drinking water, pH to 10.5 ± 0.2 with 10% (w/v) NaOH or 10% (v/v) HCl
 Extraction Disk: **ENVI-8 DSK, 47mm**
 Cat. No.: **57172**
 Conditioning:
 10 mL methanol
 2 x 10 mL water
 10 mL (0.5g cetyl trimethyl ammonium bromide/5mL ammonium hydroxide in 500mL water, diluted to 1L)
 2 x 10 mL water
 10 mL (10.0g sodium hexanesulfonic acid/10mL ammonium hydroxide in 250mL water, diluted to 500mL)
 Do not allow disk to dry between solutions
 100 mL/min flow rate
 Sample Addition:
 Extraction:
 1 mL methanol, then 2 x 4 mL (13.5 mL orthophosphoric acid/10.3 mL diethylamine in 500 mL water, diluted to 1 L)
 Column:
 C18 HPLC, 15 cm x 4.6 mm ID, 5 µm particles
 Mobile Phase:
 3.5 mL triethylamine/1.0 g sodium 1-hexanesulfonic acid in 800 mL water (pH to 2.5 with orthophosphoric acid), diluted to 1 L
 Oven:
 35°C
 Flow:
 2.0 mL/min
 Det.:
 photodiode array, 308 nm (diquat) or 257 nm (paraquat)
 Inj.:
 100 µL

For details of the extraction procedure, see US EPA Method 549.1.



794-0715

ENVI-Disk



994-0256

ENVI-Disk Holder



995-0117

ENVI-Disk Holder Manifold



994-520

ENVI-Disk Clamp



996-0280

Ordering Information:

Description	Cat. No.
ENVI-8 DSK SPE Disks	
C8 bonded phase	
47mm, pk. of 24	57172
SUPELCOSIL™ LC-18 HPLC Column	
15cm x 4.6mm ID, 5µm particles	58230-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
90mm Filter Clamp Assembly	57262
Replacement Teflon Stage, 90mm	57263

* Flask and tube not included with holder.

Reference

1. US Environmental Protection Agency Method 549.1.
Request from National Technical Information Service, U.S. Department of Commerce, Springfield, VA 22161 USA (Tel. 703-487-4650).

Trademarks

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 Teflon – E.I. du Pont de Nemours & Co., Inc.

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