



Empore™ Extraction Disks

Method Summary

EPA Method 549.1

Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and High Performance Liquid Chromatography with Ultraviolet Detection

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The complete method is available as a part of Supplement II from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703. (800) 553-6847

Summary

This method determines diquat and paraquat in drinking water and drinking water sources. Results for a waste water are also reported in the performance data. The analytes are extracted from the water using 47 mm, C8 solid phase extraction disks and ion-pairing. Final analysis uses HPLC with UV detection.

<u>Analyte</u>	<u>MDL (µg/L)</u>	<u>Mean %R*</u>	<u>%RSD</u>
Diquat	0.51	105	8
Paraquat	0.59	93	6

* Spiking level for each compound 5.0 µg/L; n=9

Method

1. A 250 ml sample is adjusted to pH 10.5 with 10% aqueous sodium hydroxide or 10% aqueous hydrochloric acid.
 2. Assemble Empore™ 47 mm C8 bonded silica extraction disks in an all-glass** or plastic 47 mm filtration apparatus. (Manifolds are appropriate for multiple samples.)
- ** Since diquat and paraquat tend to adsorb onto glass surfaces, all glassware that comes into contact with samples, sample extracts, or standard solutions should be silanized. The use of plastic labware circumvents this necessity.
3. Add 10 ml of methanol to the disk and allow to soak for about one minute. Apply vacuum to pull most of the methanol through the disk leaving a 3 to 5 mm layer of methanol on top of the disk.
 4. Add 10 ml reagent water to the disk. Using the vacuum, pull most of the water through the disk leaving 3 to 5 mm of water on the surface of the disk. Not all bonded silicas are capable of achieving acceptable recoveries in this method. The C8 silica used in Empore disks has been tested for both diquat and paraquat recoveries.

5. Apply 10 ml of conditioning solution A*** to the disk; and with vacuum, pull a small amount through then allow the disk to soak for one minute. With vacuum, draw most of the solution through the disk, leaving a 3 to 5 mm layer on top of the disk. Solution A contains cetyltrimethylammonium bromide to deactivate residual silanols.
6. Using two 10 ml aliquots of reagent water, rinse solution A from the disk. Always allow a 3 to 5 mm layer of liquid above the disk.
7. Repeat step 5 using conditioning solution B***. Solution B contains 1-hexanesulfonic acid, which bonds to the C8, forming a cation exchange sorbent.
8. Add the water sample to the reservoir and start the vacuum. Pull the sample through the disk as fast as the vacuum will allow. Drain as much of the water from the sample bottle as possible.
9. Remove the filtration assembly and insert suitable sample tube for eluate collection.
10. Add 0.5-1.0 ml of methanol to the disk and allow to soak for about one minute. Add 4 ml of the disk eluting solution*** to the disk and allow to soak for one minute. Pull most of the solution through the disk, leaving 3 to 5 mm on the disk. The disk eluting solution contains acid and diethylamine, which disrupts the ion-pair mechanism.
11. Add another 4 ml of disk eluting solution to the remaining solution on the disk, and pull it completely through.
12. Using disk eluting solution, make the eluate to a known (usually 10 ml) volume. The extract is now ready for HPLC.

*** Note: Complete details about the preparation and composition of reagent solutions can be found in Method 549.1. For the sake of brevity, these details have been omitted from this document.

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