

# CARTRIDGE FOR DIQUAT/ PARAQUAT ANALYSIS



ENVIRO-CLEAN® METHOD 549.2



FORENSICS



PHARMA

The UCT C8 cartridge (EEC08156) for Diquat (CAS 85-00-7) and Paraquat (CAS 1910-42-5) analysis is designed to provide a new level of performance in solid-phase extraction.

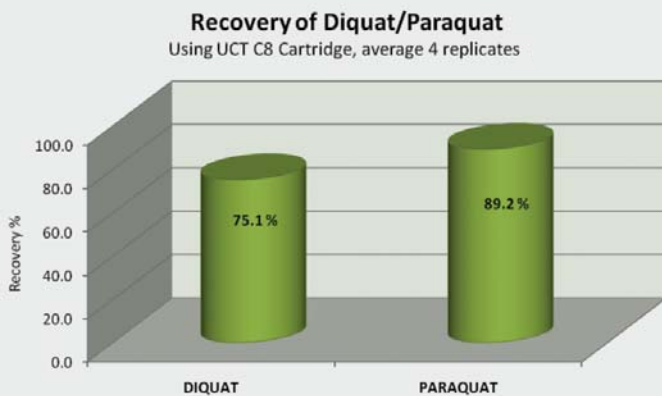
## Product Benefits

- SPE using bonded end-capped C8
- Excellent pH stability (1-14)
- Teflon frits eliminate potential contamination
- Works well at all levels of analyte loading
- No Lot to Lot variability
- Excellent reproducibility and detection (MDL Diquat 1.6 µg/L, Paraquat 1.7 µg/L)
- Packaged in metalized foil to maintain product cleanliness

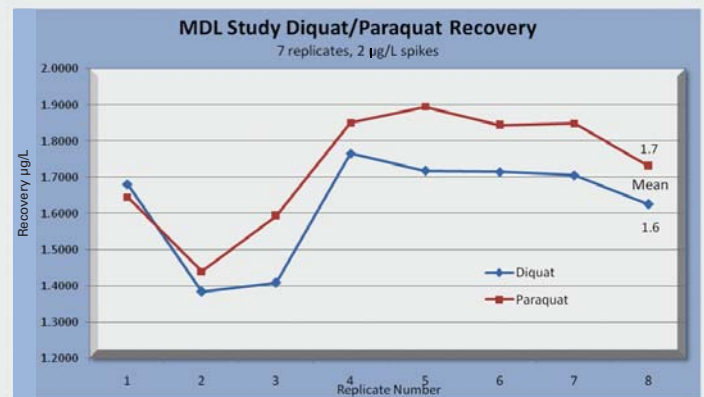
## Product Features

- 6 mL volume cartridge manufactured from special proprietary polypropylene
- Can be used on manual single or multi-station manifolds system
- Can be used with automated extraction systems

Not all sorbents are capable of achieving acceptable recoveries using method 549.2. The bonded C8 used in UCT cartridges has been tested for both diquat and paraquat recoveries.



Average Recovery of 40.0 µg/L Spikes  
in DI Water from 4 Replicates



Results of an MDL Study Using 7 Replicates  
2.0 µg/L spikes

- Mean Recovery = Diquat 1.6255 µg/L and Paraquat 1.7310 µg/L
- Standard Deviation = Diquat 0.1587 µg/L and Paraquat 0.1725 µg/L

## EPA Method 549.2

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

#### Method Summary

This method is used for the determination of diquat (1,1'-ethylene-2,2'-bipyridium dibromide salt) and paraquat (1,1'-dimethyl-4,4'-bipyridium dichloride salt) in drinking water and drinking water sources. The analytes are extracted from 250 mL of water adjusted to pH 7-9 using a 6 mL C8 solid phase extraction cartridge with ion-pairing solution. Analytes are eluted from the cartridge, separated with HPLC then detected with UV photodiode array at 308nm and 257 nm, respectively.

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#### Initial Preparation

- Since diquat and paraquat are ionic analytes there is the potential for adsorption on glass surfaces. All glassware should be silanized prior to use to deactivate the surface before use.
- The use of plastic lab ware is preferable but must be thoroughly wash before use
- Adjust a 250 mL of sample to pH 7 - 9 with 10 % aqueous sodium hydroxide or 10 % aqueous hydrochloric acid solution depending upon initial pH condition
- Assemble a C8 (UCT EEC08156) bonded silica extraction cartridge in an all-glass or plastic filtration apparatus
- If the sample contains particulates, filter through 0.45 µm Nylon membrane filter

#### Interferences

- Glassware must be scrupulously clean
- Clean all glassware with the last solvent used in it followed by detergent wash
- Heat in oven at 130°C for several hours before use
- Always use high purity solvents

#### Sample Collection, Preservation and Storage

- Grab samples must be collected in amber PVC bottles or silanized amber glass bottles
- Samples must be chilled at 4°C from collection to analysis
- Samples with residual chlorine must be preserved with sodium thiosulfate at 100 mg/L
- Samples with biologically active constituents must be preserved at pH 2 with sulfuric acid
- All samples must be extracted within 7 days of collection; extracts must be analyzed within 21 days

#### Diquat dibromide and paraquat dichloride Stock Solutions (1000 mg/L)

- Dry diquat and paraquat salts in an oven at 110°C for three hours. Cool in a desiccator
- Repeat process to a constant weight.
- Weigh 0.1968 g of dried diquat salt and 0.1770 g of dried paraquat salt

- Transfer to a silanized glass or polypropylene 100 mL volumetric flask. Add approximately 50 mL of deionized water then dilute to the mark with deionized water

#### Procedure

**Once conditioning procedure has started, do not allow cartridge to dry out otherwise repeat. Cartridge flow should be about 3 – 6 mL/minute.**

##### 1) Cartridge Conditioning

- Place cartridge(s) on a vacuum manifold
- Add 5 mL of reagent water to the cartridge
- Add 5 mL of methanol to the cartridge and soak for about one minute
- Apply vacuum to draw most of the methanol through the cartridge. Leave a thin layer on top of the frit
- Add 5 mL reagent water to the cartridge
- Apply vacuum and draw most of the water through the cartridge. Leave a thin layer of water on the frit
- Apply 5 mL of conditioning **Solution A (Note 1)** to the cartridge
- Draw a small amount through then allow the cartridge to soak for one minute leaving a thin layer on the frit
- Use 5 mL of reagent grade water to rinse the Solution A from the cartridge. Allow a thin layer of water to remain on the cartridge frit
- Repeat the above procedure using 20 mL of conditioning **Solution B.**

##### Note 2. Do not rinse

- Retain Solution B in the cartridge to keep it activated
- Cartridges may be prepared in advance and stored up to 48 hour prior to use if sealed and stored at 4°C. Do not let the cartridge dry out

##### 2) Sample Extraction

- Determine the pH of the sample. Adjust to 7.0 – 9.0 with 10% NaOH or 10% v/v HCl before extracting
- Add the 250 mL of the water sample to the reservoir and start the vacuum
- Draw the sample through the cartridge draining as much water from the sample bottle as possible
- Rinse the cartridge with 5 mL of HPLC grade methanol
- Draw vacuum through the cartridge until dry
- Remove the filtration assembly and insert a silanized 5 mL volumetric flask for collection of the eluate

##### 3) Cartridge Elution

- Add 4.5 mL of the **cartridge eluting solution (Note 3)** to the cartridge and allow to soak for one minute
- Elute at 1-2 mL per minute
- Draw any remaining solution through the cartridge. The cartridge eluting solution contains acid and diethylamine which disrupts the ion-pair interactions releasing the analytes
- Using cartridge **ion-pair solution, Note 4** add 100 µL to the flask
- Bring the eluate to a known volume of 5 mL using **cartridge eluting solution**
- The extract is now ready for HPLC analysis as shown below



## HPLC Conditions

**Column:** Phenomenex Spherisorb 3  $\mu$ , 4.6 mm x 100 mm

**Column Temperature:** 35.0°C

**Flow Rate:** 2.0 mL/minute, Ion-Pair Mobile Phase

**Injection Volume:** 200  $\mu$ L

**Photodiode Array Detector Settings:** Wavelength

**Range:** 210-370 nm

**Wavelengths:** Diquat 308 nm, Paraquat 257 nm

## Procedure Notes

**1.Solution A:** contains cetyl trimethyl ammonium bromide to deactivate residual silanol groups on the C8. Dissolve 0.500 grams cetyl trimethyl ammonium bromide and 5 mL of ammonium hydroxide in 500 mL of reagent water. Dilute to 1000 mL

**2.Solution B:** Dissolve 10 g 1-hexanesulfonic acid sodium salt and 10 mL of ammonium hydroxide in 250 mL of DI water then dilute to 500 mL. This solution adsorbs to C8 forming a cation exchange sorbent

**3.The Cartridge Elution Solution:** Dissolve 13.5 mL of orthophosphoric acid and 10.3 mL of diethylamine in 500 mL of DI water, then dilute to 1 liter

**4.Ion-pair Concentrate:** Dissolve 3.75 grams of 1-hexanesulfonic acid in 15 mL in the Cartridge Elution Solution (Note 3) and dilute to 25 mL in a volumetric flask with additional Cartridge Elution Solution

\*EPA Method 549, Issued August, 1992 as a part of "Methods for the Determination of Organic Compounds in Drinking Water," Supplement 11; Revision 1.0; Hodgeson, J.W., Bashe, W.J., (Technology Applications, Inc.) and Eichelberger, J., Environmental Monitoring Systems Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268. Complete details about the preparation and composition of reagent solutions can be found in the method at [www.epa.gov/safewater/methods/methods.html](http://www.epa.gov/safewater/methods/methods.html)

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## WARRANTY

All products manufactured by UCT are guaranteed against defects in materials and workmanship for a period of 90 days after shipment. UCT will replace any items that prove to be defective during this time period.

The exclusive remedy requires the end user to first advise UCT of the defective product by phone or in writing. Secondly, the defective product must be returned within 30 days after proper approval from our Quality Manager. All returns must indicate the purchase order number, the lot number and the shipping date. UCT's total liability is limited to the replacement cost of UCT products.

This warranty does not apply to damage resulting from misuse.

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