

Analysis of Diquat and Paraquat in Drinking Water Using EPA Method 549.2 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

EPA Method 549.2 outlines the procedure for the extraction and analysis of the herbicides, diquat and paraquat in water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 549.2 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of semi-automating these processes apparent.

To meet demands for a low cost method that requires less financial investment than fully automated systems, FMS developed a simple semi-automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC-UV

Consumables

- FMS, Inc. 0.5 g C-18 cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid or Sodium Hydroxide
- Cetyl trimethyl ammonium bromide
- Ammonium hydroxide
- 1-hexane sulfonic acid
- Orthophosphoric acid
- Fisher Pesticide Grade Methanol
- Diethyl amine

- Restek 549.2 spiking standards

Procedure

- 6 samples (250 mL water each) are prepared and adjusted with NaOH or HCl till pH ~ 7-9
- Spike with various 549.2 standards
- Put sample bottles in place and fill rinse bottles with 4.5 mL EPA 549.2 Eluting Solution
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 5 mL water, 5 mL methanol, 5 mL water, 5 mL EPA 549.2 Conditioning Solution A, 5 mL water, 10 mL methanol, 5 mL water and 20 mL EPA 549.2 Conditioning Solution B.
- Samples are loaded across cartridges under vacuum, at 3-6 mL/min.
- Cartridges are dried under vacuum for 1 min (no nitrogen).
- Sample bottles are automatically rinsed from the rinse bottles with 4.5 mL EPA 549.2 Eluting Solution.

Stage 2:

- EPA 549.2 Eluting Solution from sample bottles is loaded across the cartridges (2 x 10 mL, 1 min soak), under vacuum at 1-2 mL/min and the eluent is collected for analysis into Direct to LC Vial Collection Vessels, and fortified with EPA 549.2 Ion-pair Concentrate.
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from C-18 cartridges

FMS SuperVap®

- Pre-heat temp: 45 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 45 °C under nitrogen (7-10 psi)
- Direct to LC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

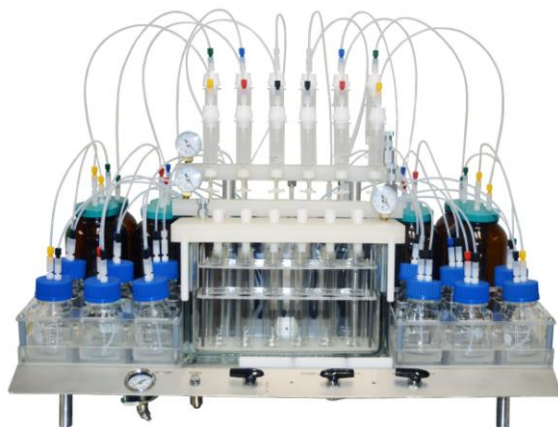


Table 1 with recoveries for the Method 549.2 analytes

Compound	%Recovery	Stdev
Diquat	92.7	3.3
Paraquat	84.2	4.9

Conclusions

We repeatedly obtained high recoveries and close precision for both diquat and paraquat, demonstrating excellent efficiency for these analytes. Samples can be taken from collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



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FMS EZSpe® System