



## Featured Application: Paraquat and Diquat on Raptor HILIC-Si

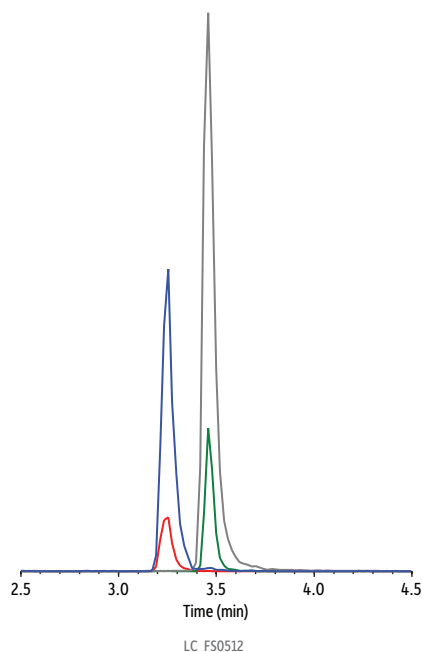
# LC-MS/MS Analysis of Paraquat and Diquat without Ion-Pairing Reagents

- Good retention and peak shape without system contamination from ion-pairing reagents.
- Consistent resolution and response with MS-friendly mobile phases.
- Fast, 7-minute gradient method supports high sample throughput.

Paraquat is a fast-acting, nonselective contact herbicide that is relatively inexpensive. These characteristics have led to its widespread use on agricultural crops in much of the developing world. While paraquat is reasonably safe to apply with proper precautions, accidental or deliberate ingestion has an extremely high fatality rate. Largely for this reason, paraquat has been restricted in many countries. Diquat is a related herbicide that is often formulated with paraquat and is monitored due to its potential toxic effects on the central nervous system. LC-MS/MS analysis of paraquat and diquat in low-moisture food crops, drinking water, and surface water is an important tool for preventing or limiting human and environmental exposure.

Typically, LC-MS/MS analysis of paraquat and diquat involves the use of ion-pairing reagents in order to achieve adequate retention, resolution, and peak shape. However, ion-pairing reagents are known to compete with target analytes for ionization, which typically results in reduced sensitivity. These reagents can also contaminate the LC-MS/MS system, requiring that it be taken off-line frequently for extensive cleaning.

Here, we have developed an LC-MS/MS analysis of paraquat and diquat that does not use ion-pairing reagents, but still provides good retention and peak shape. Both target analytes were fully resolved and showed much greater sensitivity at 50 ng/mL (50 ppb) compared to ion-pairing methods. This fast, 7-minute analysis provided consistent MS results using a 50 mM buffered mobile phase, which is within the buffer salt concentration limits that are generally recommended to minimize contamination and maintenance for LC-MS/MS systems.



| Peaks       | t <sub>R</sub> (min) | Precursor Ion 1 | Product Ion 1 | Precursor Ion 2 | Product Ion 2 |
|-------------|----------------------|-----------------|---------------|-----------------|---------------|
| 1. Diquat   | 3.25                 | 183.3           | 157.2         | 183.3           | 130.1         |
| 2. Paraquat | 3.46                 | 185.3           | 170.3         | 171.3           | 77.2          |

|                       |                                                                    |
|-----------------------|--------------------------------------------------------------------|
| <b>Column</b>         | Raptor HILIC-Si (cat.# 9310A52)                                    |
| <b>Dimensions:</b>    | 50 mm x 2.1 mm ID                                                  |
| <b>Particle Size:</b> | 2.7 µm                                                             |
| <b>Pore Size:</b>     | 90 Å                                                               |
| <b>Temp.:</b>         | 45 °C                                                              |
| <b>Sample</b>         | Paraquat & diquat calibration mix (cat.# 32437)                    |
| <b>Diluent:</b>       | Mobile phase B                                                     |
| <b>Conc.:</b>         | 50 ng/mL (cat.# 32437 diluted to 50 ng/mL in mobile phase B)       |
| <b>Inj. Vol.:</b>     | 5 µL                                                               |
| <b>Mobile Phase</b>   |                                                                    |
| A:                    | Water, 50 mM ammonium formate, 0.5% formic acid                    |
| B:                    | 25:75 Water:acetonitrile, 50 mM ammonium formate, 0.5% formic acid |

| Time (min) | Flow (mL/min) | %A | %B  |
|------------|---------------|----|-----|
| 0.00       | 0.6           | 0  | 100 |
| 4.00       | 0.6           | 35 | 65  |
| 4.01       | 0.6           | 0  | 100 |
| 7.00       | 0.6           | 0  | 100 |

|                   |       |
|-------------------|-------|
| <b>Detector</b>   | MS/MS |
| <b>Ion Mode:</b>  | ESI+  |
| <b>Mode:</b>      | MRM   |
| <b>Instrument</b> | HPLC  |

## Raptor HILIC-Si LC Columns (USP L3)



| Length                | 2.1 mm<br>cat.# | 3.0 mm<br>cat.# | 4.6 mm<br>cat.# |
|-----------------------|-----------------|-----------------|-----------------|
| <b>2.7 µm Columns</b> |                 |                 |                 |
| 30 mm                 | 9310A32         |                 |                 |
| 50 mm                 | 9310A52         | 9310A5E         | 9310A55         |
| 100 mm                | 9310A12         | 9310A1E         | 9310A15         |
| 150 mm                | 9310A62         | 9310A6E         | 9310A65         |

## EXP Reusable Fittings for HPLC & UHPLC

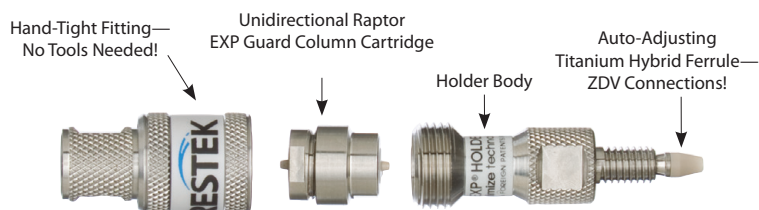
for 10-32 fittings and 1/16" tubing  
EXP Hand-Tight Fittings



| Description                            | qty.   | cat.# |
|----------------------------------------|--------|-------|
| EXP Hand-Tight Fitting (Nut w/Ferrule) | ea.    | 25937 |
| EXP Hand-Tight Fitting (Nut w/Ferrule) | 10-pk. | 25938 |
| EXP Hand-Tight Nut (w/o Ferrule)       | ea.    | 25939 |

Hybrid Ferrule U.S. Patent No. 8201854, EXP Holders U.S. Patent No. 8696902, EXP2 Wrench U.S. Patent No. D766055. Other U.S. and Foreign Patents Pending. The EXP, Free-Turn, and the Opti- prefix are registered trademarks of Optimize Technologies, Inc.

## Raptor EXP Guard Cartridges



Protect your investment and extend the life of our already-rugged LC columns and change guard column cartridges by hand without breaking fluid connections—no tools needed!

### EXP Direct Connect Holder

| Description                                                                                 | qty. | cat.# |
|---------------------------------------------------------------------------------------------|------|-------|
| EXP Direct Connect Holder for EXP Guard Cartridges (includes hex-head fitting & 2 ferrules) | ea.  | 25808 |

Maximum holder pressure: 20,000 psi (1,400 bar)

### Raptor EXP Guard Column Cartridges

| Description                                | Particle Size | qty.  | 5 x 2.1 mm<br>cat.# | 5 x 3.0 mm<br>cat.# | 5 x 4.6 mm<br>cat.# |
|--------------------------------------------|---------------|-------|---------------------|---------------------|---------------------|
| Raptor HILIC-Si EXP Guard Column Cartridge | 2.7 µm        | 3-pk. | 9310A0252           | 9310A0253           | 9310A0250           |

Maximum cartridge pressure: 600 bar/8,700 psi (2.7 µm) or 400 bar/5,800 psi (5 µm).  
Raptor SPP LC columns combine the speed of SPP with the resolution of USLC technology.  
Learn more at [www.restek.com/raptor](http://www.restek.com/raptor)

Hybrid Ferrule U.S. Patent No. 8201854, EXP Holders U.S. Patent No. 8696902, EXP2 Wrench U.S. Patent No. D766055. Other U.S. and Foreign Patents Pending. The EXP, Free-Turn, and the Opti- prefix are registered trademarks of Optimize Technologies, Inc.

## Reference Standards

### Paraquat & Diquat Calibration Mix (2 components)

Diquat dibromide (6385-62-2)  
Paraquat dichloride (1910-42-5)

1,000 µg/mL each in water, 1 mL/ampul cat.# 32437 (ea.)

