

PARAQUAT & DIQUAT

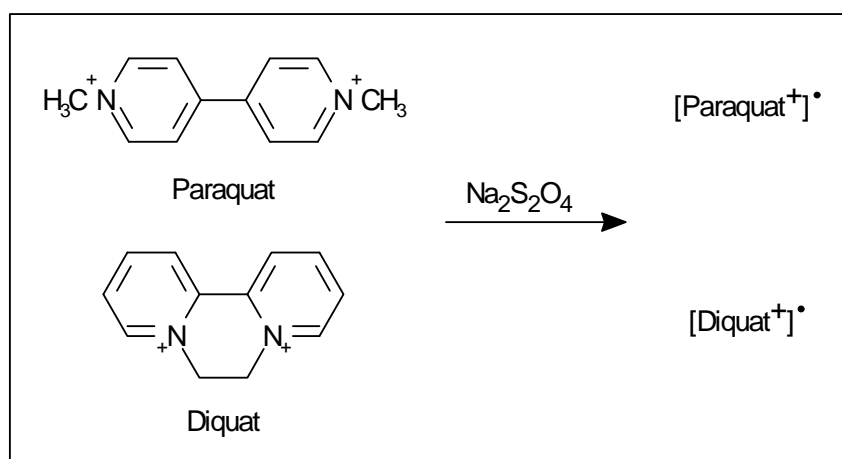


Paraquat and diquat are used as non-selective herbicides for ground and water plants. Actually, it was paraquat, which was used to destroy the marijuana crop in Mexico. Paraquat & diquat tend to accumulate in soil and water due to their high resistance and their polar structure (quaternary ammonium salt). Since both have a highly toxic effect on humans, a precise determination of their concentration is of great importance.

In order to detect traces of paraquat and diquat, PICKERING offers their one-step post-column derivatization system PINNACLE PCX. Pickering has developed an application kit for this application, which consists of the ALKION™-column, guard column, and eluants. The great advantage of the Pickering ion-exchange column lies in the simple sample preparation.

Description of the Method

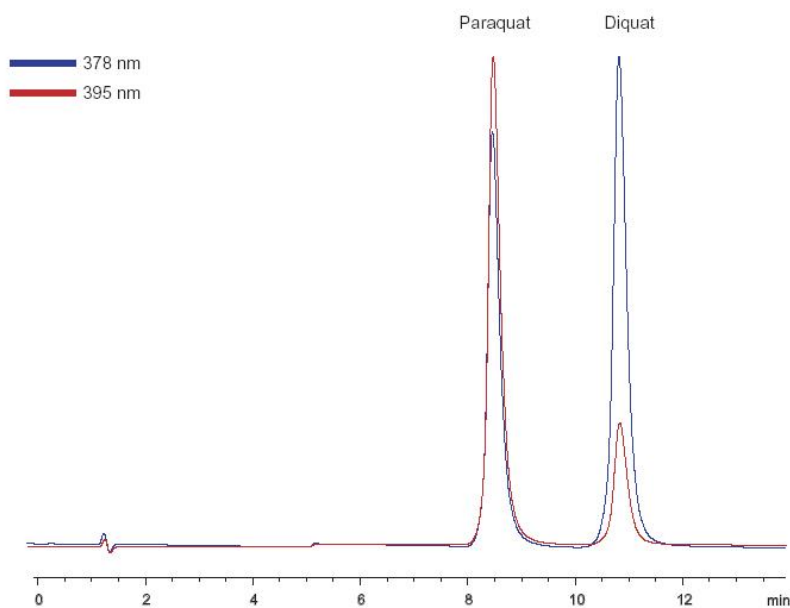
The one-step derivatization of paraquat and diquat is performed by reduction with alkaline sodium dithionite solution resulting in strongly UV-absorbing, coloured radicals.



APPLICATION NOTE

Chromatogram

Chromatogram of a Spiked Water Sample



HPLC-Terms and Derivatization Parameter

HPLC	
Operation Mode	Quaternary gradient
Eluant	1. Potassium phosphate/borate buffer (0.1 M, pH 6.0) 2. Potassium hydroxide solution (0.1 M, pH 13.0) 3. Potassium chloride solution (1.0 M, pH 6.0) 4. iso-Propanol
Degassing	Helium- or vacuum degassed
HPLC Column	ALKION™-column 4 x 150 mm
Flow Rate	0.8 mL/min

APPLICATION NOTE

Pinnacle PCX	Single-pump; 150 µL reactor
Column Oven	40 °C
Reactor Volume	150 µL
Reactor Temperature	RT
Reagent	0,3 % Sodium dithionit in 0,3 N sodium hydroxide (w/v)
Reagent Flow	0.3 mL/min
Detection	
Detection Type	UV/VIS Detection
Paraquat	395 nm
Diquat	378 nm
Flowcell	Analytic; pressure stable up to 7 bar

Caution: Extreme pH-range! (Only valid for Pickering method)

As a consequence of the alkaline regenerant (pH 13), components made from Vespel may not be present in the HPLC system, instead these must be exchanged for components made from pH-inert materials (Tefzel or PEEK). For advice, please contact your LC-representative or consult your technical manual.

An inert version (Titanium, PEEK) of the HPLC system is not necessary, a piston seal wash for the pump heads, however, is recommended.

To avoid corrosion of the system and contamination of the ion-exchange column with metal ions, passivating is recommended; this is especially valid for older systems. For advice, please contact your LC-representative or consult your technical manual.

APPLICATION NOTE

Gradient Program

METHOD „Paraquat & Diquat“: ALKION®-Ion-Exchange Column

Flow rate: 0,8 mL/min; column temperature: 50°C

Step	Time [min]	Interval [min]	K01 [%]	K02 [%]	K03 [%]	i-PrOH [%]	
1	0	-	31.5	53.5	0	15	Injection
2	10	10	28.0	47.0	10	15	Linear gradient
3	15	5	28.0	47.0	10	15	Isocratic elution
4	15.1	0.1	31.5	53.5	0	15	Step change
5	15.1	5 - 9	31.5	53.5	0	15	Re-equilibration

Literature

PICKERING LABORATORIES, *Application Note 106*.

V. A. Simon, *LC GC 1987*, 5, 899 – 903.

Chemicals and Columns

Post-Column Derivatization Unit

Order Number	Description
1153-1012	PINNACLE PCX; single-pump, 150 mL reactor

Application Kit

Order Number	Description
0352-0041	Application Kit for the analysis of paraquat & diquat, consists of:
9410917	ALKION®, Cation-exchange column, K ⁺ -form, 4 x 150 mm
9493020	Guard column for ALKION™ column, 3 x 20 mm
1100-0265	Restrictor
1700-1101	Potassium phosphate/borate buffer, pH 6,0, 4 x 950 mL
1700-1102	Potassium hydroxide solution, 4 x 950 mL
1700-1103	Potassium chloride solution, 4 x 950 mL

APPLICATION NOTE

Cation-Exchange Columns for Paraquat & Diquat Analysis

Each column for the analysis of paraquat & diquat is tested by applying an calibration standard and running it according to a specific gradient elution protocol. After a column passes this rigorous QC test, it receives a serial number and is packaged with its test chromatogram.

Order Number	Description
9410917	ALKION™, Cation-exchange column, K ⁺ -form, 4 x 150 mm
9493020	Guard column for ALKION™ column, 3 x 20 mm

Potassium Phosphate Buffer

- 1) Filtered, ready to use
- 2) Free from fluorescent impurities
- 3) Cost-effective: long shelf life; can be used to the last ml
- 4) Consistent elution profiles, bottle to bottle, lot to lot

These buffers are used for the elution of paraquat and diquat according to the method developed by Pickering.

Order Number	Description
1700-1101	Potassium phosphate/borate buffer, pH 6.0, 4 x 950 mL
1700-1102	Potassium hydroxide solution, 4 x 950 mL
1700-1103	Potassium chloride solution, 4 x 950 mL

Restrictor

In order to get a higher operating pressure in the HPLC, a restrictor can be install

Order Number	Description
1100-0265	Restrictor; 300 psi at 0.3 mL/min; 10 cm