Analysis of paraquat and diquat by CE/MS

Application Note

Environmental

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Abstract

Paraquat is one of the most widely used herbicides in the world. This type of chemical herbicide –bipyridyl– is shared with the related chemical, diquat. Both of these compounds are highly toxic. Paraquat has severe and irreversible delayed effects where death can occurr up to 30 days after ingestion. Both capillary electrophoresis (CE) and ion-pair reversed phase LC have been used for the analysis of these compounds with CE considered simpler and cheaper[1]. Here we describe a method for the analysis of these compounds using CE coupled to electrospray ionization mass spectrometry (ESI/MS).

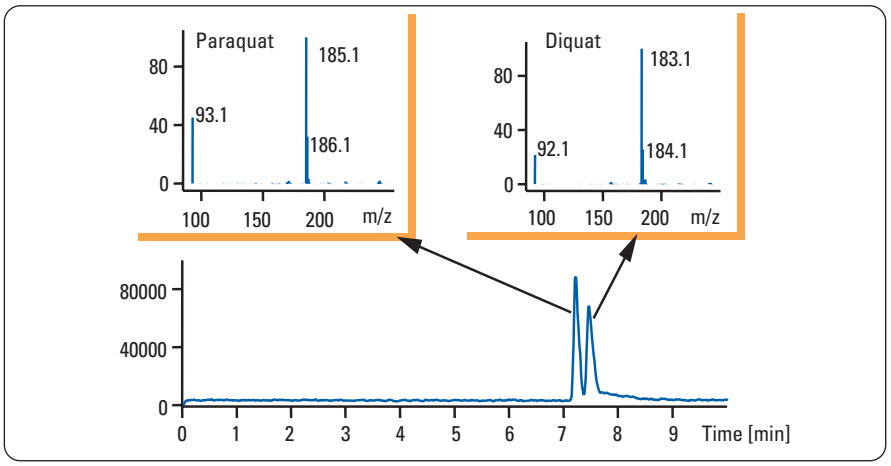

Figure 1
Total ion electropherogram and mass spectra for paraquat and diquat (100 µg/mL, scan 90 to 250 *m/z*).

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Experimental

CE-ESI/MS analysis was performed using the Agilent Capillary Electrophoresis system coupled to the Agilent MSD equipped with electrospray source and orthogonal sprayer for CE/MS. Sheath liquid was delivered by an Agilent binary pump equipped with a 1:100 flow splitter. The Agilent ChemStation software was used for instrument control.

Results

The CE/MS method was capable of quickly separating and unequivocally identifying paraquat and diquat (figure 1). The assay has a reproducibility for migration time for paraquat and diquat of 0.14 and 0.16 % respectively and for peak areas of 4.5 and 4.2 %. The assay was linear over the range 0 to 10 mg/ml with r^2 of 0.99928 and 0.99956 for paraquat and diquat respectively. Limits of detection could be greatly improved using selected ion monitoring (SIM) and large volume injections (figure 2). Under these conditions the limits of detection were 100 ng/mL giving a signal-to-noise of 7.3 for paraquat and 5.7 for diquat. The assay is suitable for detection of paraquat and diquat poisoning in contaminated foods and beverages, such as coffee (figure 3).

Reference

1.
Carniero MC, Puignou L and Galceran MT. *J Chromatogr. 669, 217-224,* **1994.**

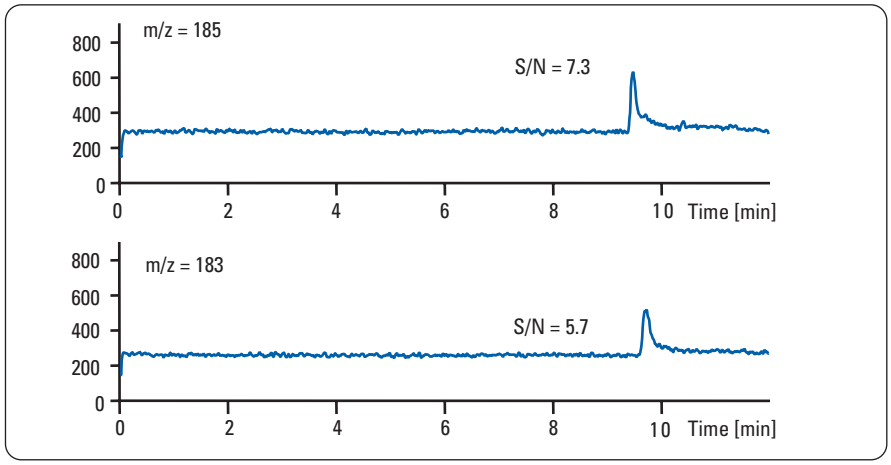

Figure 2
SIM of paraquat and diquat using large volume injection.

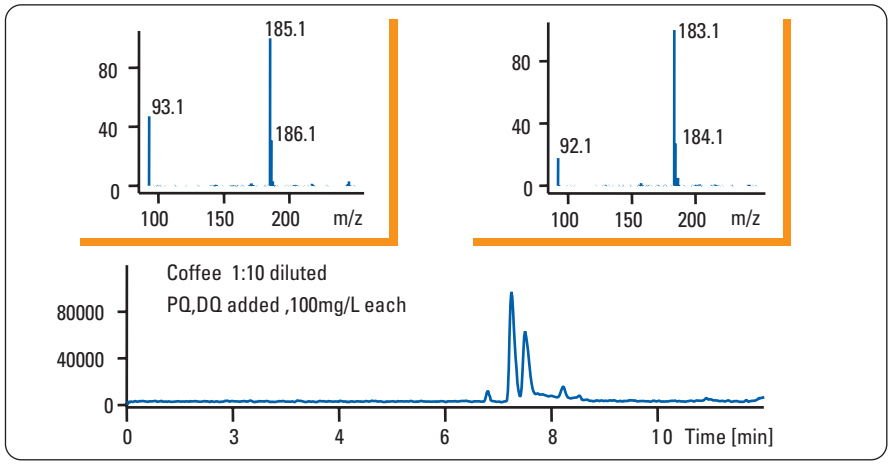

Figure 3
Analysis of paraquat and diquat in coffee.

Chromatographic conditions

CE

Buffer:	50 mM ammonium acetate (pH 4.0) in 50 % methanol
Injection:	300 mbar · seconds
Capillary:	PVA coated 75 µm x (21.6 cm UV) 78 cm MS
Voltage:	30 kV
Temperature:	20 °C

MS

Sheath liquid:	5 mM ammonium acetate in 50 % methanol
Flow rate:	10 µL/min
Polarity:	positive
Capillary voltage:	4000 V
Fragmentor:	60 V
Gain:	1
Drying gas:	N_2
Drying gas temp:	300 °C
Nebulizer gas pressure:	10 psi
Scan:	90 to 250 *m/z* or SIM 185.1 *m/z* and 183.1 *m/z*

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