

Analysis of drugs of abuse by CE-ESI-MS

Application Note

Drug Testing

Authors

Dat T. Phan
Peter B. Harrsch
Agilent Technologies,
Waldbronn, Germany

Abstract

Samples of drug substances containing LSD and a mixture of methamphetamine, cocaine, and heroin, were analyzed by capillary electrophoresis coupled with electrospray ionization mass spectrometry (CE-ESI-MS). Both protonated molecular ions and characteristic fragment ions were observed, allowing unique identification. The data shown here demonstrate feasibility. Additional method development and/or validation may be required for routine use.

Experimental

The Agilent CE system with CE-MS capillary cassette was connected to an Agilent MSD. The MSD was fitted with electrospray ionization and orthogonal sprayer for CE-ESI-MS. Sheath liquid was delivered by an Agilent binary pump equipped with a 1:100 flow splitter. All instrumentation was controlled by an Agilent ChemStation. The samples were provided by a law enforcement agency specifically for these studies. Stock solutions were made in methanol and then diluted with water. The fused silica capillary was prepared by flushing for 30 minutes with 1 N NaOH followed by a 10-minute wash with deionized water prior to insertion into the sprayer needle. Once installed the capillary was flushed with running buffer for 30 minutes.



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Results

Figure 1A shows the analysis of LSD. The mass spectrum of the peak at approximately 15 minutes reveals the protonated molecular ion at 324.2 m/z. Ions with masses of 223.1 and 281.2 are characteristic fragment ions caused by collision induced dissociation (CID). At fragmentor values greater than 100 V another characteristic fragment ion at a mass of 208.2 appears (data not shown). Based on these observations, a very specific method for identification of LSD could be developed. The total ion electropherogram (TIE) in Figure 1B shows the separation of a mixture of methamphetamine, cocaine and heroin. In the mass spectrum of the peak at 12.5 minutes the ion with mass 150.1 is the protonated molecular ion of methamphetamine. The ions at m/z 119.1 and 91.1 are fragment ions and are in greater abundance than the molecular ion. Methamphetamine undergoes CID at lower fragmentor voltages and hence is significantly fragmented at the 100 V used for this analysis. In this experiment no fragment ions are observed for cocaine or heroin, only the protonated molecular ions at 304.1 and 370.1 m/z, respectively (mass spectra not shown). To obtain confirmatory fragmentation, and add specificity to the method for these two compounds, the fragmentor voltage would have to be increased. Alternatively a fragmentor ramp from 100 V to higher values would give fragmentation patterns for cocaine and heroin. CE provides a selectivity that allows the analysis of these compounds using a single method. In combination with spectra and fragmentation patterns obtained from MS detection, this provides a rapid and unequivocal identification of these compounds in drug seizures.

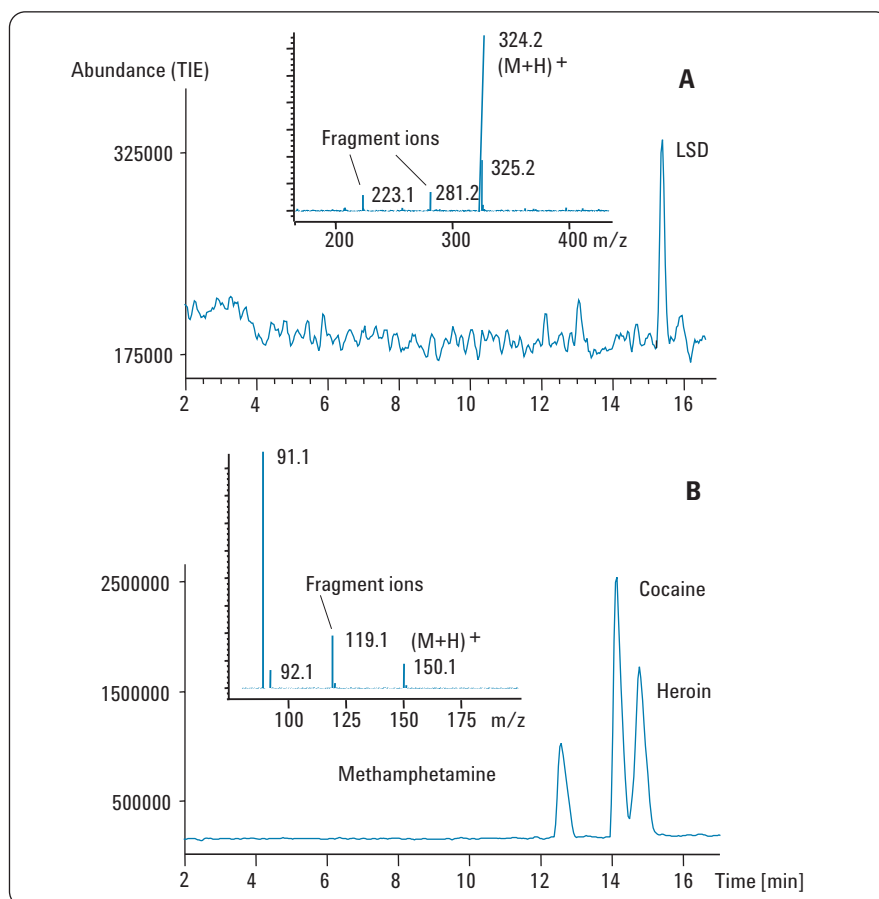


Figure 1
Total ion electropherograms and mass spectra of LSD and a mixture of methamphetamine, cocaine and heroin.

Chromatographic conditions

Sample: A) LSD (8 µg/mL), B) heroin, methamphetamine and cocaine (300 µg/mL)
Injection: 3 sec @ 50 mbar
Capillary: bare fused silica, total length 100 cm, 50 µm id
Buffer: 50 mM ammonium acetate, pH 4.5
Voltage: 25 kV
Temperature: 25°C
Preconditioning: 3 min flush with buffer at 1 bar
Sheath liquid: 0.5 % acetic acid in 50 % acetonitrile, 5 µL/min
Nebulizing gas: nitrogen, 20 psi
Drying gas: nitrogen, 7 L/min, 200 °C
Acquisition: positive mode, Vcap -3.5 kV, fragmentor 100 V, step size 0.1, PW 0.25 min, time filter on
Scan range: 80–500 m/z

Equipment

- Agilent Capillary Electrophoresis system
- Agilent CE-MS Adapter Kit
- Agilent LC/MSD module with API Electrospray Source
- Agilent CE-ESI-MS Sprayer Kit
- Agilent ChemStation and CE-MS software

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Published March 1, 2009
Publication Number 5990-3394EN



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