



High Resolution, Accurate Identification of Small Molecule Unknowns with UPLC and the maXis

Bruker Daltonics Inc.

Modern analytical applications often demand definitive tandem MS results on ever more complex samples utilizing fast separation techniques. maXis™ is the only mass spectrometer able to deliver the maximum MS performance specification at the very highest speeds delivered by modern Ultra Performance Liquid Chromatography (UPLC) and Capillary Electrophoresis (CE). The maXis is specially designed to deliver excellent results in many applications including:

- Small Molecule Identification
- Impurity and Degradent Identification
- In vitro and in vivo Drug Metabolite Identification
- Intact Protein Analysis
- Quantitative Proteomics and Protein Identification

Redefining High Performance Mass Spectrometry

With resolution in excess of 40,000 and MS and MS-MS mass accuracy typically between 600–800 ppb at speeds of up to 20 full spectra per s simultaneously. No other mass spectrometer is better equipped to deliver definitive data on complex samples in proteomics, metabolomics, and small molecule identification challenges.

Typical results from the maXis:

- 20 Hz speed of acquisition at high resolution for high-speed chromatography
- 40 k+ resolution in both MS and MS-MS mode
- Wide dynamic range of 5 orders of magnitude for trace detection in complex mixtures
- Sub-ppm mass accuracy in both MS and MS-MS mode for high confidence IDs

Experimental

Sample: Club Drug Mix of 10 Small Molecule Drugs

UPLC System

Waters Acquity UPLC with DAD detector (210–400 nm)
Column: Acquity BEH C18 1.7 μ m 2.1 \times 50 mm
Column Temperature: 40 °C

Mobile Phase

- A) 0.1% Formic Acid in Water
- B) 0.1% Formic Acid in ACN

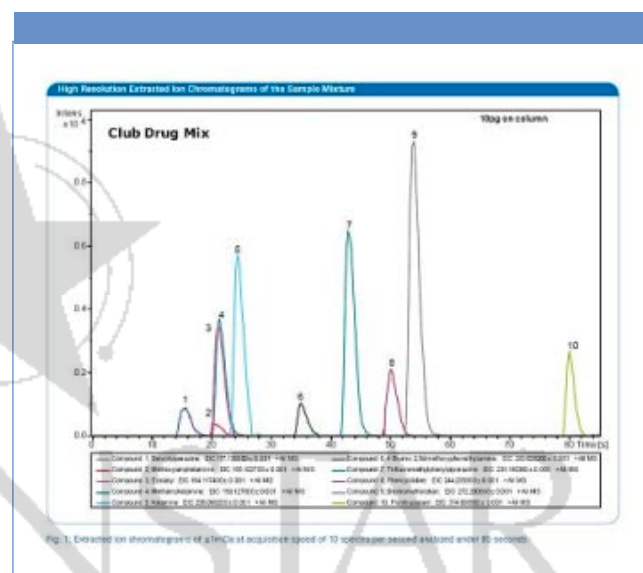


Figure 1: Extracted ion chromatograms of ± 1 mDa at acquisition speed of 10 spectra per s analyzed under 85 s.

Gradient

20%–40% B in 60 s
40%–100% B in 5 s
100% B for 172 s *followed by column equilibration
Flow Rate: 0.6 mL/min
Injection volume: 2 μ L

MS System

Bruker Daltonics maXis
Spectral Acquisition 10 full spectra per s
Dry Heater 180 °C
Nebulizer 2 Bar
Capillary 4500 V
Dry Gas 6 L/min
ESI positive polarity
Range 100–800 m/z
Calibrant Lithium Formate (16mM)
LockMass Calibration Methylstearate

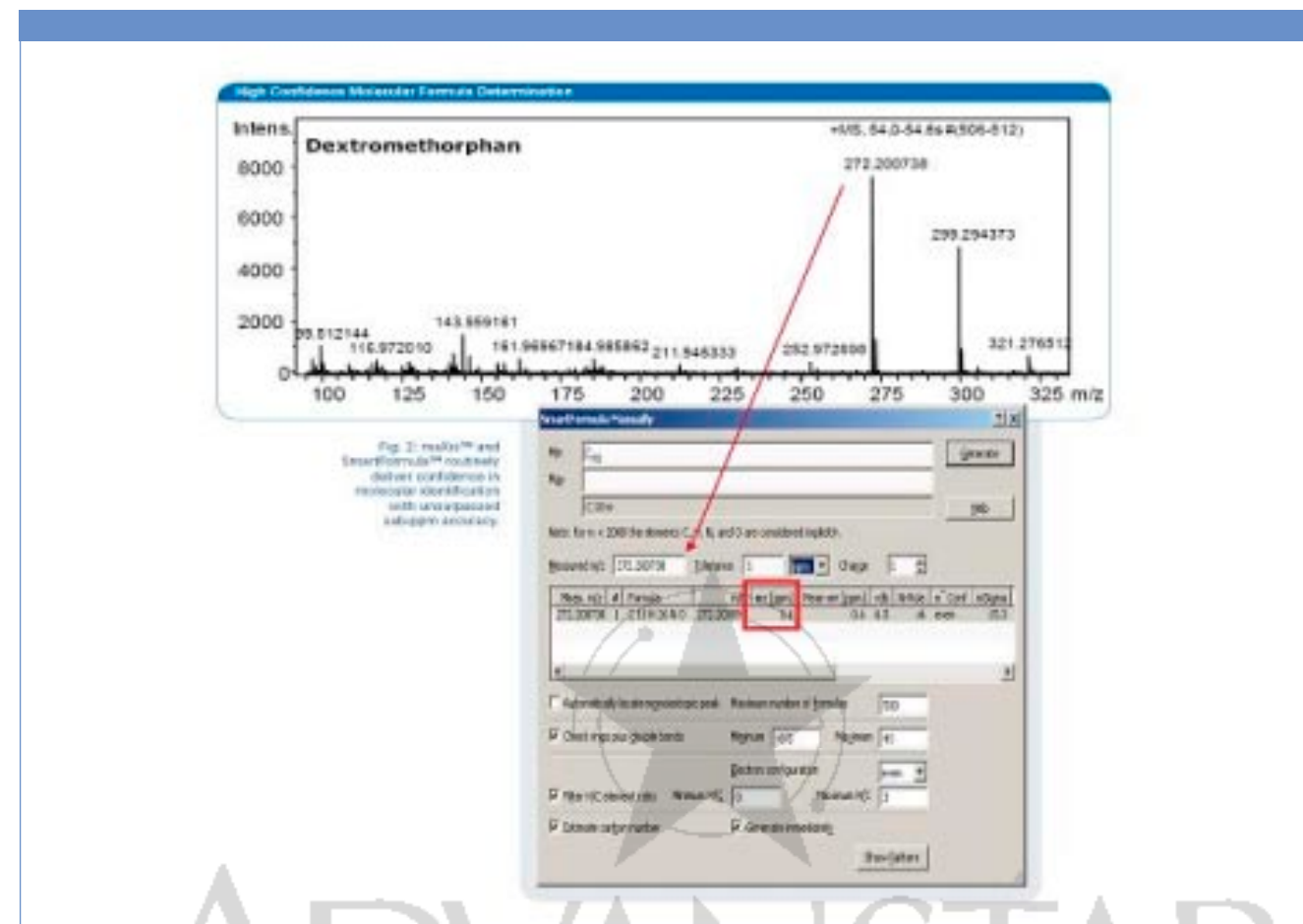


Figure 2: maXis™ and SmartFormula™ routinely deliver confidence in molecular identification with unsurpassed sub-ppm accuracy.

Results and Conclusion

In this study, the maXis was challenged to identify a series of unknown drug compounds that had been separated by UPLC. Using a typical reverse phase C18 gradient separation method, 7 of the 10 compounds were base line separated by the UPLC in 85 s at 10 Hz sampling rate. The maXis was able to discern the presence of 10 compounds in the sample, despite the lack of chromatographic separation of three of the compounds, even at levels as low as 10 pg on column. Furthermore, the excellent mass accuracy (<1 ppm) and resolution (Avg ~50 K) of the results generated by the maXis, when used in conjunction with the SmartFormula 3D software, were able to correctly identify all 10 compounds and unequivocally determine their molecular formula. This is exceptional performance from a qTOF, especially with a UPLC speed separation.

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