

Application Note No. 083

The Large Volume Aqueous Injection of a Parent Amino Acid Extract

Diane Nicholas

- **Monitors breakdown products of sample at high temperature**
- **Removes need for pre-concentration of water extract**
- **No derivatisation steps needed prior to analysis**

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- ATAS Optic 2-200 programmable injector
 - Agilent 5890 gas chromatograph with 5972 mass selective detector

Principles

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- Extract the parent amino acid with water
 - Inject 10 μL of water extract onto packed liner and vent solvent
 - Switch to splitless mode and rapidly heat injector to high final temperature to breakdown parent amino acid
 - Monitor breakdown products by GC-MS

Chromatogram

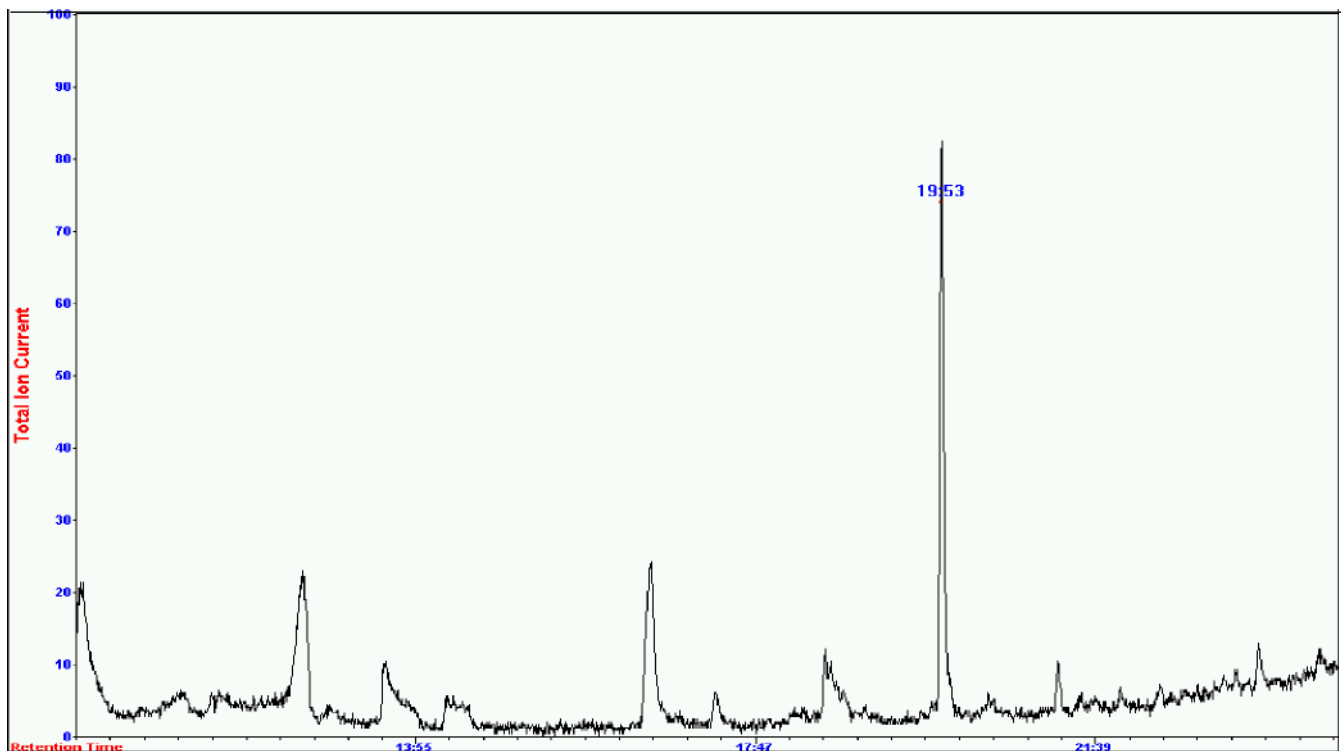


Figure: Breakdown product (t_r 19.53) produced from a 10 μL injection of sample containing 0.8 ng of parent amino acid collected in SIM mode

Current limit of detection: 80 $\text{pg}/\mu\text{L}$ of parent amino acid

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Appendix

Optic Parameters:

Liner: 'A' Type
Mode: Large volume
Gas Flows: Vent: 300 mL/min
Split: 30 mL/min
Initial temperature: 100 °C
Vent time: 7 mins
Ramp rate: 16 °C/s
Final temperature: 300 °C
End time: 60 mins
Split open time: 3 mins
Purge pressure: 8 psi
Transfer pressure: 12.6 psi
Transfer time: 2 mins
Initial pressure: 12.6 psi
Final pressure: 12.6 psi

Volume injected: 10 µL
Extraction solvent: Water

GC Parameters:

Column: HP Innowax 30m x 0.25 mm i.d. x 0.25 µm film
Initial temperature: 40 °C

Initial time: 10 mins 15
Ramp rate 1: °C/min 80
Final temperature 1: °C
Ramp rate 2: 10 °C/min
Final temperature 2: 240 °C
Final time: 10 mins

MS Parameters:

Acquisition mode: SIM
Transfer line: 280 °C