

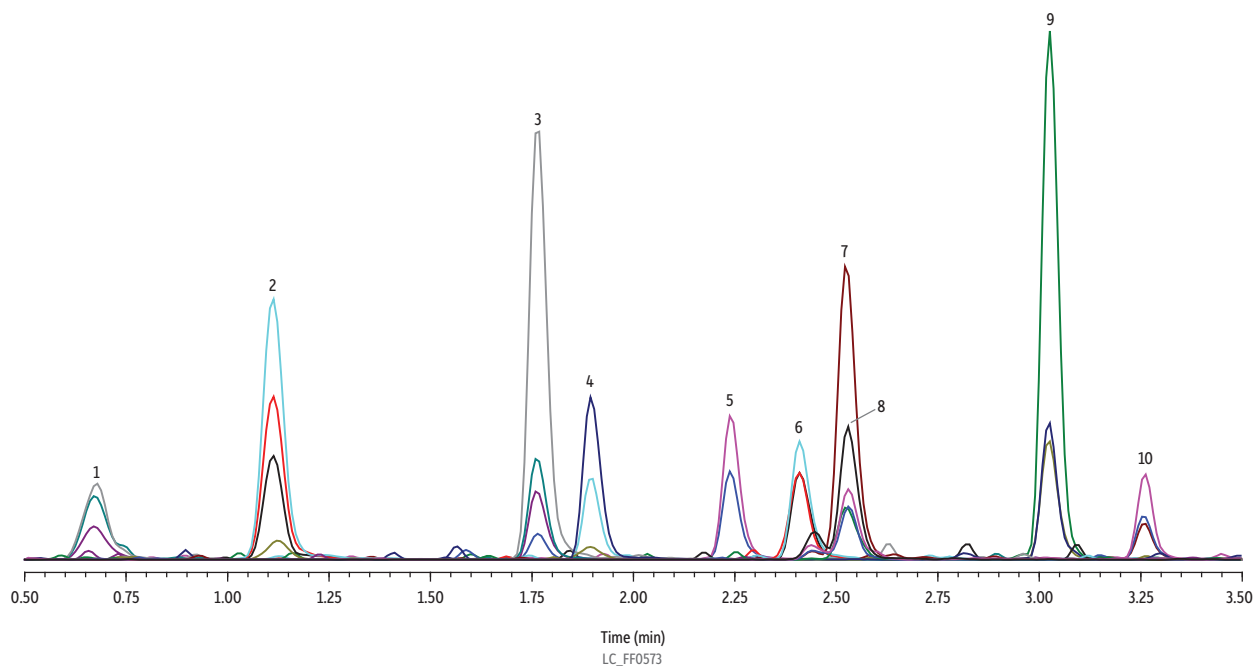


Featured Application: Pyrrolizidine Alkaloids on Raptor ARC-18

Rapid, Simple 4.5-Minute Pyrrolizidine Alkaloids Analysis by LC-MS/MS

- Fast 4.5-min pyrrolizidine alkaloids analysis separates most compounds to baseline.
- Simple mobile phase and gradient elutes compounds quickly with excellent peak shape.
- Robust Raptor ARC-18 column outlasts traditional C18 columns under acidic conditions.

Pyrrolizidine alkaloids (PAs) are toxins found naturally in a wide variety of plant species (e.g., daisies, forget-me-nots, and legumes, among others). They are secondary metabolites formed to protect plants against herbivores and, according to the European Food Safety Authority (EFSA), are likely the most widely distributed natural toxins. While there are over 6000 plant species known to contain pyrrolizidine alkaloids, only a few species have been involved in the direct poisoning of people or animals. And, of the roughly 700 known PAs, the 1,2-unsaturated compounds have been identified as the most toxic, capable of causing fatal liver damage. Herbs, teas, honey, and cereal may be contaminated with pyrrolizidine alkaloids if plants containing PAs are accidentally co-harvested or used as a food source for bees.



Peaks	tx (min)	Precursor	Product 1	Product 2	Product 3
1. Monocrotaline	0.677	326.20	120.3	121.30	94.25
2. Incidine	1.111	300.20	94.15	138.20	156.36
3. Heliotrine	1.763	314.30	138.20	156.20	120.20
4. Heliotrine N-oxide	1.895	330.25	172.25	138.20	
5. Senecionine	2.240	336.20	120.30	138.30	
6. Senecionine N-oxide	2.410	352.35	94.20	120.30	118.25
7. Echimidine	2.524	398.30	120.25	220.15	
8. Echimidine N-oxide	2.528	414.25	396.30	352.20	254.25
9. Lasiocarpine	3.028	412.20	120.20	220.15	336.25
10. Lasiocarpine N-oxide	3.260	428.25	254.25	352.25	120.30

Column: Raptor ARC-18 (cat.# 9314A52); Dimensions: 50 mm x 2.1 mm ID; Particle Size: 2.7 µm; Pore Size: 90 Å; Temp.: 25 °C; **Sample:** Diluent: 0.1% Formic acid in water; Conc.: 10 ng/mL; Inj. Vol.: 1 µL; **Mobile Phase:** A: 0.1% Formic acid in water, B: 0.1% Formic acid in acetonitrile; **Gradient (%B):** 0.00 min (5% B), 3.5 min (35% B), 3.51 min (5% B), 4.5 (5% B); **Flow:** 0.5 mL/min; **Detector:** MS/MS; Ion Mode: ESI+; **Instrument:** UHPLC.

Because of the World Health Organization's (WHO) Codex Alimentarius Commission adoption of the "as low as reasonably achievable" (ALARA) principle for PAs—along with many regional efforts to monitor these toxic compounds in food, feed, and medicines, the need for fast, accurate pyrrolizidine alkaloids analysis methods is on the rise. At present, the only harmonized method was developed and validated by the German Federal Institute for Risk Assessment (BfR) [1]. It employs a sulfuric acid digestion, an SPE C18 cleanup, and analysis via LC-MS/MS. The example method provided in the BfR protocol uses a 150 x 2.1 mm, 1.9 μ m fully porous particle C18 column and produces a run time of 15 minutes with the last compound, lasiocarpine-N-oxide (LaN), eluting at 9.33 minutes. However, in the case study presented below, switching to a superficially porous 50 x 2.1 mm, 2.7 μ m Raptor ARC-18 column provides sufficient retention while still eluting LaN in just 3.2 minutes (4.5 minute total run time). Note that although a UHPLC instrument was used in this example, it is not necessary for this analysis. Excellent peak shapes were obtained and all compounds, except for echimidine and its corresponding N-oxide, were separated to baseline. The Raptor ARC-18 column's unique phase chemistry also results in longer lifetimes under acidic conditions compared to traditional C18 columns.

As the scope of pyrrolizidine alkaloids analysis continues to expand, methods will have to grow along with it, but for today's challenges, using a Raptor ARC-18 column allows labs to optimize method conditions, shorten the overall run time, and increase sample throughput.

References

[1] Bundesinstitut für Risikobewertung (BfR), BfR-PA-Tee-2.0/2014, Bestimmung von Pyrrolizidinalkaloiden (PA) in Pflanzenmaterial mittels SPE-LC-MS/MS, 2014. <https://www.bfr.bund.de/cm/343/bestimmung-von-pyrrolizidinalkaloiden.pdf>



Column Characteristics:

Stationary Phase Category: C18, octadecylsilane (L1)
 Ligand Type: Sterically protected C18
 Particle: 1.8 μ m, 2.7 μ m, or 5 μ m superficially porous silica (SPP or "core-shell")
 Pore Size: 90 Å
 Carbon Load: 7% (1.8 μ m), 7% (2.7 μ m), 5% (5 μ m)
 End-Cap: no
 Surface Area: 125 m²/g (1.8 μ m), 130 m²/g (2.7 μ m), or 100 m²/g (5 μ m)
 Recommended Usage:
 pH Range: 1.0–8.0
 Maximum Temperature: 80 °C
 Maximum Pressure: 1,034 bar/15,000 psi* (1.8 μ m), 600 bar/8,700 psi (2.7 μ m); 400 bar/5,800 psi (5 μ m)



* For maximum lifetime, recommended maximum pressure for 1.8 μ m particles is 830 bar/12,000 psi.

Properties:

- Well-balanced retention profile.
- Sterically protected and acid-resistant to resist harsh, low-pH mobile phases.
- Ideal for use with sensitive detectors like mass spec.

Switch to an ARC-18 when:

- You are analyzing large, multiclass lists by LC-MS/MS.
- Strongly acidic (pH 1–3) mobile phases are required.

Raptor ARC-18 LC Columns (USP L1)

Chromatographic Properties

Designed and intended specifically for use on LC-MS/MS systems, the Raptor ARC-18 column offers a well-balanced retention profile without the drawbacks of using an ordinary C18 in the harsh, acidic mobile phases needed for mass spectrometry (MS). Even after extended use in these low-pH (≤ 2.0) conditions, the sterically protected ARC-18 offers consistent retention, peak shape, and response for charged bases, neutral acids, small polar compounds, and more. For the rapid analysis of large, multiclass assays by LC-MS/MS, the acid-resistant Raptor ARC-18 truly is *ahead of the curve*.

Length	2.1 mm cat.#	3.0 mm cat.#	4.6 mm cat.#
1.8 μm Columns			
30 mm	9314232	—	—
50 mm	9314252	931425E	—
100 mm	9314212	931421E	—
150 mm	9314262	—	—
2.7 μm Columns			
30 mm	9314A32	9314A3E	9314A35
50 mm	9314A52	9314A5E	9314A55
100 mm	9314A12	9314A1E	9314A15
150 mm	9314A62	9314A6E	9314A65
5 μm Columns			
30 mm	—	931453E	—
50 mm	9314552	931455E	9314555
100 mm	9314512	931451E	9314515
150 mm	9314562	931456E	9314565
250 mm	—	—	9314575