Featured Application: *Ergot Alkaloid Mycotoxins on Raptor Biphenyl*

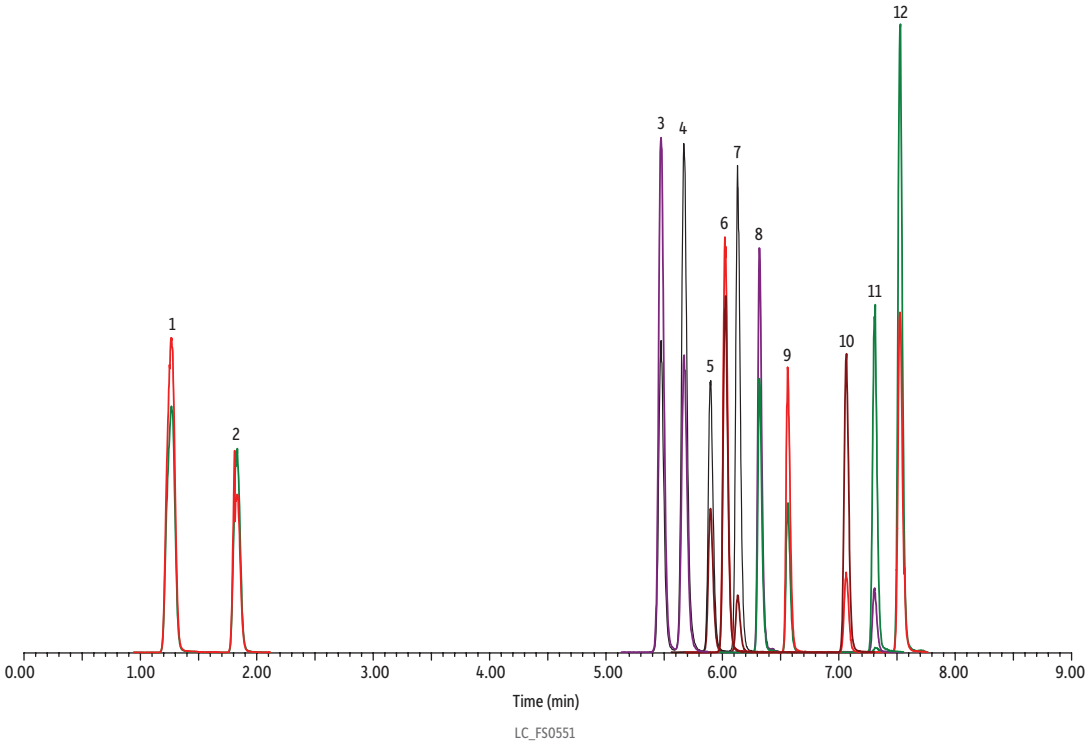
Analysis of Ergot Alkaloid Mycotoxins in Blended Flour by LC-MS/MS Under Acidic Conditions

- Baseline separation of six critical ergot alkaloids and their epimers allows definitive quantification.
- Fast, 11-min total cycle time supports high-throughput testing.
- Acidic conditions suitable for the simultaneous analysis of ergot alkaloids, *Alternaria* toxins, and major regulated mycotoxins.

Ergot alkaloids are mycotoxin contaminants of cereal plants that are of increasing regulatory interest due to their widespread presence and the dietary importance of cereals. Of more than 40 known ergot alkaloids, six compounds, including ergocornine, ergocristine, ergocryptine, ergometrine, ergosine, and ergotamine, together with their isomeric -inine epimers, are currently regulated by the EU. When these ergot alkaloids are analyzed on a C18 column, high pH conditions are needed to separate all 12 epimers. In contrast, when a Raptor Biphenyl column is used, baseline separation can be achieved under regular column- and MS-friendly, acidic conditions. As shown here, complete resolution of all 12 epimers, including the challenging ergotamine/ergotaminine and ergosine/ergosinine epimer pairs, was obtained in 11 minutes of total cycle time.

The use of a Raptor Biphenyl column and acidic conditions also allows concurrent analysis of ergot alkaloids with other mycotoxins of interest. A separate study established that these conditions are suitable for quantitative analysis of ergot alkaloids in the same run as *Alternaria* toxins and priority regulated mycotoxins in a variety of foods [1]. Quantitation in that study was performed in wheat baby cereal, peanut, tomato puree, and blended flour using matrix-matched standards because isotopically labeled standards were not available for these mycotoxins. Suitability of this method for multiresidue mycotoxin testing provides food safety labs with a more efficient workflow and the time-saving benefit of combined analysis.

Figure 1: Baseline separation of all ergot alkaloid epimers in a blended flour sample was achieved on a Raptor Biphenyl column under acidic conditions.



Peaks	t_R (min)	Precursor Ion	Product Ion 1	Product Ion 2
1. Ergometrine	1.27	326.2	223.2	208.1
2. Ergometrinine	1.83	326.2	223.2	208.1
3. Ergosine	5.47	548.4	208.1	223.2
4. Ergosinine	5.67	548.4	208.1	223.2
5. Ergotamine	5.90	582.4	223.2	268.2
6. Ergocornine	6.03	562.4	268.2	223.2
7. Ergotaminine	6.13	582.4	223.2	268.2
8. Ergocryptine	6.32	576.4	268.2	223.2
9. Ergocristine	6.56	610.4	223.2	592.4
10. Ergocorninine	7.07	562.4	268.2	223.2
11. Ergocryptinine	7.31	576.4	268.2	223.2
12. Ergocristinine	7.53	610.4	223.2	592.4

Column Raptor Biphenyl (cat.# 9309A12)
Dimensions: 100 mm x 2.1 mm ID
Particle Size: 2.7 µm
Pore Size: 90 Å
Guard Column: Raptor Biphenyl EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9309A0252)
Temp.: 60 °C
Standard/Sample
Diluent: 50:50 Water:methanol
Conc.: 6.25 ng/mL final concentration after sample preparation
Inj. Vol.: 5 µL
Mobile Phase
A: Water, 0.05% formic acid
B: Methanol, 0.05% formic acid

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	75	25
5.00	0.4	50	50
9.00	0.4	0	100
9.01	0.4	75	25
11.0	0.4	75	25

Detector MS/MS
Ion Mode: ESI+
Mode: MRM
Instrument UHPLC
Sample Preparation A blended flour was prepared by mixing white rice flour (75%); brown rice flour (5%); millet flour (5%); oat flour (5%); all-purpose wheat flour (5%); and all-purpose, gluten-free flour (5%). Two grams of the flour sample were weighed into a 50-mL polypropylene centrifuge tube (cat.# 25846) and fortified at 50 µg/kg for all analytes with a stock standard solution. After sitting at room temperature for 10 minutes, 16 mL of extraction solution (80:20 acetonitrile:water) containing 0.5% formic acid were added, and the tube was stirred to create a homogenous suspension. The extraction was carried out by shaking horizontally on a digital pulse mixer (Glas-Col LLC, Terre Haute, IN) at 800 rpm for 20 minutes. After centrifuging for 5 minutes at 4000 rpm, 1 mL of extract was evaporated to dryness at 45 °C under a gentle stream of nitrogen. The dried extract was reconstituted with 1 mL of 50:50 water:methanol solution, and a 0.4 mL aliquot was transferred to and filtered using a Thomson SINGLE StEP filter vial with a 0.2 µm PTFE filter (cat.# 25874). Five µL of the filtered solution was injected for the LC-MS/MS analysis.

Notes Want even better performance when analyzing metal-sensitive compounds? Check out Inert LC columns at www.restek.com/inert

References

1. S.H. Liang, J. York, J. Konschnik, H. Majer, J. Steimling, Comprehensive mycotoxin analysis: simultaneous determination of *Alternaria* toxins, ergot alkaloid epimers, and other major mycotoxins in various food matrices by LC-MS/MS, Restek Corporation, 2022.

Featured Products

Analytical Column	Sample Handling	Maintenance & Accessories
		
Raptor Biphenyl LC columns (cat.# 9309A12) **Raptor EXP guard column cartridges** (cat.# 9309A0252)	**Thomson SINGLE StEP eXtreme filter vials** (cat.# 25874)	**Waste overflow indicator for LC systems** (cat.# 26543 & 26550)

Want even better performance when analyzing mycotoxins and other metal-sensitive compounds?

Learn more at **www.restek.com/inert**

ordering notes

Certificates of analysis for new Restek LC columns are now provided electronically. To view and download, visit www.restek.com/documentation then enter your cat.# and serial #.

Want even better performance when analyzing mycotoxins? Check out Inert LC columns at **www.restek.com/inert**

Raptor Biphenyl LC Columns (USP L11)

- Ideal for bioanalytical testing applications like drug and metabolite analyses.
- Heightened selectivity and retention for compounds that are hard to resolve or elute early on C18 and other phenyl chemistries.
- Limits ionization suppression and allows simple, MS-friendly mobile phases.
- Part of Restek's Raptor LC column line featuring 1.8, 2.7, and 5 µm SPP core-shell silica.

Stationary Phase Category: Phenyl (L11)
Ligand Type: Biphenyl
Particle: 1.8 µm, 2.7 µm, or 5 µm superficially porous particle (SPP or "core-shell" particle) silica
Pore Size: 90 Å
Carbon Load: 7% (1.8 µm); 7% (2.7 µm); 5% (5 µm)
End-Cap: yes
Surface Area: 125 m^2/g (1.8 µm); 130 m^2/g (2.7 µm); or 100 m^2/g (5 µm)
Recommended Usage:
pH Range: 2.0 to 8.0
Maximum Temperature: 80 °C
Maximum Pressure: 1034 bar/15,000 psi* (1.8 µm); 600 bar/8700 psi (2.7 µm); 400 bar/5800 psi (5 µm)

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

ID	Length	qty.	cat.#
2.7 µm Particles Raptor Biphenyl			
2.1 mm	100 mm	ea.	9309A12

Raptor Inert Biphenyl HPLC Columns

- Inert LC column technology reduces nonspecific binding of chelating analytes, enabling sensitive analysis and smooth integration of peaks.
- Ideal for the analysis of metal-sensitive compounds, such as mycotoxins.
- Increased response and analyte recovery, allowing lower detection limits.
- Improved peak shape without additional passivation or mobile phase additives.
- Part of Restek's Raptor Biphenyl column line featuring 2.7 µm SPP core-shell silica.

ID	Length	Particle Size	Units	Cat.#
2.1 mm	100	2.7 µm	ea.	9309A12-T

Raptor EXP Guard Column Cartridges

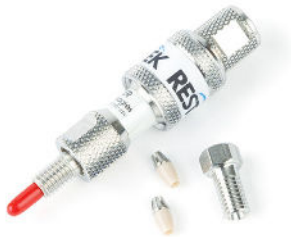
- Free-Turn architecture lets you change cartridges by hand without breaking inlet/outlet fluid connections—no tools needed.
- Patented titanium hybrid ferrules can be installed repeatedly without compromising high-pressure seal.
- Auto-adjusting design provides ZDV (zero dead volume) connection to any 10-32 female port.
- Guard column cartridges require EXP direct connect holder (cat.# 25808).

Description	Particle Size	Size	qty.	cat.#
Raptor Biphenyl EXP Guard Column Cartridge	2.7 µm	5 x 2.1 mm	3-pk.	9309A0252

Maximum cartridge pressure: 1034 bar/15,000 psi* (UHPLC); 600 bar/8700 psi (2.7 µm); 400 bar/5800 psi (5 µm).
* For maximum lifetime, recommended maximum pressure for UHPLC particles is 830 bar/12,000 psi.
Intellectual Property: optimizetech.com/patents

EXP Direct Connect Holder

A Restek LC guard cartridge in an EXP direct connect holder is the ultimate in column protection, especially when using dilute-and-shoot or other minimal sample preparation techniques.



Description	qty.	cat.#
EXP Direct Connect Holder for EXP Guard Cartridges (includes hex-head fitting & 2 ferrules)	ea.	25808

Maximum holder pressure: 20,000 psi (1400 bar)
Intellectual Property: optimizetech.com/patents

Thomson SINGLE StEP eXtreme Filter Vials

Description	Color	Porosity	qty.	cat.#
Nylon				
Thomson SINGLE StEP eXtreme Filter Vials	black preslit cap	0.2 µm	100-pk.	25878
	pink preslit cap	0.45 µm	100-pk.	25879
PES (polyethersulfone)				
Thomson SINGLE StEP eXtreme Filter Vials	grey preslit cap	0.2 µm	100-pk.	25880
PTFE (polytetrafluoroethylene)				
Thomson SINGLE StEP eXtreme Filter Vials	green preslit cap	0.2 µm	100-pk.	25874
	blue preslit cap	0.45 µm	100-pk.	25875
PVDF (polyvinyldifluoride)				
Thomson SINGLE StEP eXtreme Filter Vials	red preslit cap	0.2 µm	100-pk.	25876
	yellow preslit cap	0.45 µm	100-pk.	25877

Q-sep Multispeed Centrifuge for QuEChERS

- Program 10 custom cycles for time, braking, and speed or g-force (up to 4500 rpm or 3450 xg).
- QuEChERS-specific presets for AOAC and EN methods make consistent operation quick and simple.
- Convenient lid lighting indicates at a glance if unit is ready, running, or done.
- Control panel can be temporarily locked on one cycle for error-free reproducibility.
- Cool-Flow design prevents samples from overheating by maintaining unit at room temperature.
- Tube holders are carbon fiber for high strength, durability, and years of trouble-free use.
- Clear lid permits safe observation of samples and optical calibration of speed.

Specifications

Tube Capacity	6 x 50 mL tubes 18 x 15 mL tubes 24 x 2 mL tubes
Dimensions (H x W x D)	9 in x 14.5 in x 17 in (23 cm x 37 cm x 43 cm)
Weight	39 lb (17 kg)
Sound Level	64 dB A
Environmental Range	16–32 °C
Voltage	95–253 VAC
Frequency	50/60 Hz
Power Requirement	220 Watts
Centrifuge Motor	½ H.P. Brushless DC
Max g-Force	3450 xg
Max Speed	4500 RPM
Cycle Time	30 sec to 99 min, 59 sec (±2%)

Intended Use

General-purpose laboratory centrifuge intended for safe and rapid density-based separation of fluids, including physiologic fluids, in approved specimen receptacles for qualitative or quantitative test procedures. As a general-purpose laboratory centrifuge, it is designed to also run other approved containers filled with chemicals (nonflammable, nonexplosive, nonvolatile, and non-highly reactive only), environmental samples, and other nonhuman body samples. This device is intended to be operated by properly trained personnel who have carefully read the operating manual and are familiar with the function of the device.

Description	Certification/Compliance	qty.	cat.#
Q-sep Multispeed Centrifuge for QuEChERS Includes: 15 mL four-place tube holder (6); 50 mL single-place tube holder (6); 50 mL conical tube insert (6); 2 mL tube adaptors (24); U.S. power cord (1); global/universal power cord (1)	UL61010-1/CSA C22.2 No. 61010-1 and IEC61010-2-020; FDA listed; MET U.S. E112532; CE; RoHS	ea.	28295

Empty Centrifuge Tubes, Polypropylene

Description	qty.	cat.#
Empty 50 mL Centrifuge Tube, Polypropylene w/Cap	50-pk.	25846
	500-pk.	28290 NEW!

Waste Overflow Indicator for LC Systems

Description	Certification/Compliance	qty.	cat.#
Waste Overflow Indicator for LC Systems, 4 Liter	CE	ea.	26543
Waste Overflow Indicator for LC Systems, GL-45	CE	ea.	26550

Bottle not included.

Sidewinder LC Column Heater

NEW!

- Easy to set up!
- Operation range: 5 °C above ambient to 85 °C, ±1 °C.
- Lightweight, compact design fits in small spaces.

Description	Certification/Compliance	qty.	cat.#
Sidewinder Column Heater Temp. Control Module w/Long Column Holder (25 cm)	CE	ea.	25732
Sidewinder Column Heater Temp. Control Module w/Short Column Holder (10 cm)	CE	ea.	25733

EXP2 TI-LOK All-in-One (AIO) Reusable Fittings for HPLC & UHPLC

NEW!

for 10-32 fittings and 1/16" tubing

- Titanium nut with integrated PEEK ferrule simplifies installation—one-piece design means no more lost ferrules.
- Convenient, built-in driver makes secure installations quick and easy.
- Allows easy 18,000 psi (1250 bar) seals without wrenches.
- Can be installed repeatedly without compromising high-pressure seal.

25745

Description	qty.	cat.#
EXP2 TI-LOK All-in-One (AIO) Hand-Tight Fitting with Integral Ferrule (includes built-in driver)	ea.	25745
	10-pk.	25746

Intellectual Property: optimizetech.com/patents

LC Stainless-Steel Capillary Tubing

- 316-grade stainless steel.
- Precise precut lengths.
- Clean and smooth surface finish.
- Color coded for easy identification.
- Tight tolerance: OD and ID +/-0.001".

Whether you need to replace system tubing as part of your troubleshooting or want to reduce the dwell volume of your system as you move to narrower columns, Restek has the quality tubing in the lengths and IDs you need. Each ID is color coded, so it is easy to identify and replace correctly. Tubing is precision cut, resulting in clean, square-cut ends without ovality.

Description	Color	ID	OD	Length	Max Pressure	qty.	cat.#
LC Capillary Tubing	Red	0.005" (0.127 mm)	1/16"	5 cm	27,850 psi	3-pk.	25813
	Red	0.005" (0.127 mm)	1/16"	10 cm	27,850 psi	3-pk.	25814
	Red	0.005" (0.127 mm)	1/16"	20 cm	27,850 psi	3-pk.	25815
	Red	0.005" (0.127 mm)	1/16"	30 cm	27,850 psi	3-pk.	25816
	Yellow	0.007" (0.178 mm)	1/16"	5 cm	26,610 psi	3-pk.	25817
	Yellow	0.007" (0.178 mm)	1/16"	10 cm	26,610 psi	3-pk.	25818
	Yellow	0.007" (0.178 mm)	1/16"	20 cm	26,610 psi	3-pk.	25819
	Yellow	0.007" (0.178 mm)	1/16"	30 cm	26,610 psi	3-pk.	25820
	Blue	0.010" (0.254 mm)	1/16"	5 cm	25,160 psi	3-pk.	25821
	Blue	0.010" (0.254 mm)	1/16"	10 cm	25,160 psi	3-pk.	25822
	Blue	0.010" (0.254 mm)	1/16"	20 cm	25,160 psi	3-pk.	25823
	Blue	0.010" (0.254 mm)	1/16"	30 cm	25,160 psi	3-pk.	25824
	Orange	0.020" (0.508 mm)	1/16"	5 cm	20,230 psi	3-pk.	25825
	Orange	0.020" (0.508 mm)	1/16"	10 cm	20,230 psi	3-pk.	25826
	Orange	0.020" (0.508 mm)	1/16"	20 cm	20,230 psi	3-pk.	25827
	Orange	0.020" (0.508 mm)	1/16"	30 cm	20,230 psi	3-pk.	25828

Tubing Dressing Tool

Opens stainless-steel tubing bore and removes burrs.

Description	qty.	cat.#
Tubing Dressing Tool	ea.	20188
	ea.	20190

20188

Replacement Inserts for Tubing Dressing Tool

Description	Size	qty.	cat.#
Replacement Insert	for 1/16" Tubing Dressing Tool	ea.	20189
	for 1/8" Tubing Dressing Tool	ea.	20191

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Questions? Contact us or your local Restek representative (www.restek.com/contact-us).

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