

Featured Application: Analysis of Melamine and Related Compounds on Raptor HILIC-Si

Trace-Level Analysis of Melamine and Related Compounds by LC-MS/MS

- Improve productivity—analyze five potential food adulterants in just one injection.
- Ensure accurate trace-level results with excellent sensitivity at 25 ppb.
- Increase sample throughput with a fast, 3.5-minute separation.

Foods with a high protein content command a higher price on the market, which can result in the illegal practice of food adulteration using nitrogen-rich compounds to make the protein content appear higher than the actual value. The Kjeldahl and Dumas methods are often used to measure protein content, but both methods assess protein content indirectly by measuring nitrogen content. As long as protein content is not measured directly, food adulteration with nitrogen-rich compounds for economic gain may persist, and analytical methods to detect potential adulterants (nonprotein nitrogen sources) need to be kept current, as the chemical space for cheap nitrogen-rich compounds is vast.

Melamine is a nitrogen-rich compound that has been previously identified as a food adulterant in both pet food and infant formula. Melamine can also form in vivo as a metabolite of the insecticide cyromazine. The analysis of melamine and related compounds (cyanuric acid, ammelide, and ammeline) is required when exporting feed materials to the U.S. and EU due to concerns about potential toxicity. In addition, the FDA requires that at-risk pharmaceutical components (albumin, colloidal oatmeal, gelatin, lactose, etc.) be screened for melamine before they are released for use in manufacturing or preparation of drug products. The FDA has set the tolerable daily intake level at 0.2 mg/kg for adults with the additional requirement that a single food product may only contain a maximum concentration of 2.5 ppm (1 ppm for infant formula).

The LC-MS/MS method shown here allows the combined analysis of melamine and related compounds, which is much more efficient for labs screening for multiple potential adulterants. A Raptor HILIC-Si column was selected for this method because it provides good retention of these highly polar analytes that can be difficult to retain on other phases, allowing accurate identification and quantification. Using this method, labs can analyze melamine and related compounds at residual levels with a fast separation time of only 3.5 minutes and a complete cycle time of 8 minutes. This fast, multi-analyte method is recommended for highthroughput labs that need to test for multiple potential contaminants at trace levels with quick turnaround times.





Column: Raptor HILIC-Si (cat.# 9310A52); Dimensions: 50 mm x 2.1 mm ID; Particle Size: 2.7 µm; Pore Size: 90 Å; Temp.: 30 °C; Sample: Diluent: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; Conc.: 25 ng/mL; Inj. Vol.: 5 µL Mobile Phase; A: Water, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1% formic acid; B: 5:95 Water:acetonitrile, 10 mM ammonium formate, 0.1%



Pure Chromatography

www.restek.com

Raptor HILIC-Si LC Columns (USP L3)

| RESTEK | KEZLEK |
|--------|--------|
| | |

| | Z.1 mm | 3.0 mm | 4.0 mm | |
|----------------|---------------|---------|---------|--|
| Length | cat.# | cat.# | cat.# | |
| 2.7 µm Columns | | | | |
| 30 mm | 9310A32 | | | |
| 50 mm | 9310A52 | 9310A5E | 9310A55 | |
| 100 mm | 9310A12 | 9310A1E | 9310A15 | |
| 150 mm | 9310A62 | 9310A6E | 9310A65 | |

EXP Reusable Fittings for HPLC & UHPLC

T

| for 10-32 fittings and ¹ /16" tubing | |
|---|-----|
| EXP Hand-Tight Fittings | 100 |

| Description | qty. | cat.# |
|--|--------|-------|
| EXP Hand-Tight Fitting (Nut w/Ferrule) | ea. | 25937 |
| EXP Hand-Tight Fitting (Nut w/Ferrule) | 10-pk. | 25938 |
| EXP Hand-Tight Nut (w/o Ferrule) | ea. | 25939 |

Hybrid Ferrule U.S. Patent No. 8201854, EXP Holders U.S. Patent No. 8696902, EXP2 Wrench U.S. Patent No. D766055. Other U.S. and Foreign Patents Pending. The EXP, Free-Turn, and the Opti- prefix are registered trademarks of Optimize Technologies, Inc.

Raptor EXP Guard Cartridges



Protect your investment and extend the life of our already-rugged LC columns and change guard column cartridges by hand without breaking fluid connections—no tools needed!

EXP Direct Connect Holder

| 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 (1,400 bar) | | |

Raptor EXP Guard Column Cartridges

| Description | Particle Size | qty. | 5 x 2.1 mm cat.# | 5 x 3.0 mm cat.# | 5 x 4.6 mm cat.# |
|--|------------------|-------|---------------------|---------------------|---------------------|
| Raptor HILIC-Si EXP Guard Column Cartridge | 2.7 µm | 3-pk. | 9310A0252 | 9310A0253 | 9310A0250 |

Maximum cartridge pressure: 600 bar/8,700 psi (2.7 μm) or 400 bar/5,800 psi (5 μm). Raptor SPP LC columns combine the speed of SPP with the resolution of USLC technology. Learn more at www.restek.com/raptor

Hybrid Ferrule U.S. Patent No. 8201854, EXP Holders U.S. Patent No. 8696902, EXP2 Wrench U.S. Patent No. D766055. Other U.S. and Foreign Patents Pending. The EXP, Free-Turn, and the Opti- prefix are registered trademarks of Optimize Technologies, Inc.

Stock Reference Standards

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

Melamine Stock Standard

Melamine (108-78-1) 1,000 µg/mL in diethylamine:water (20:80), 1 mL/ampul cat.# 33247 (ea.)





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