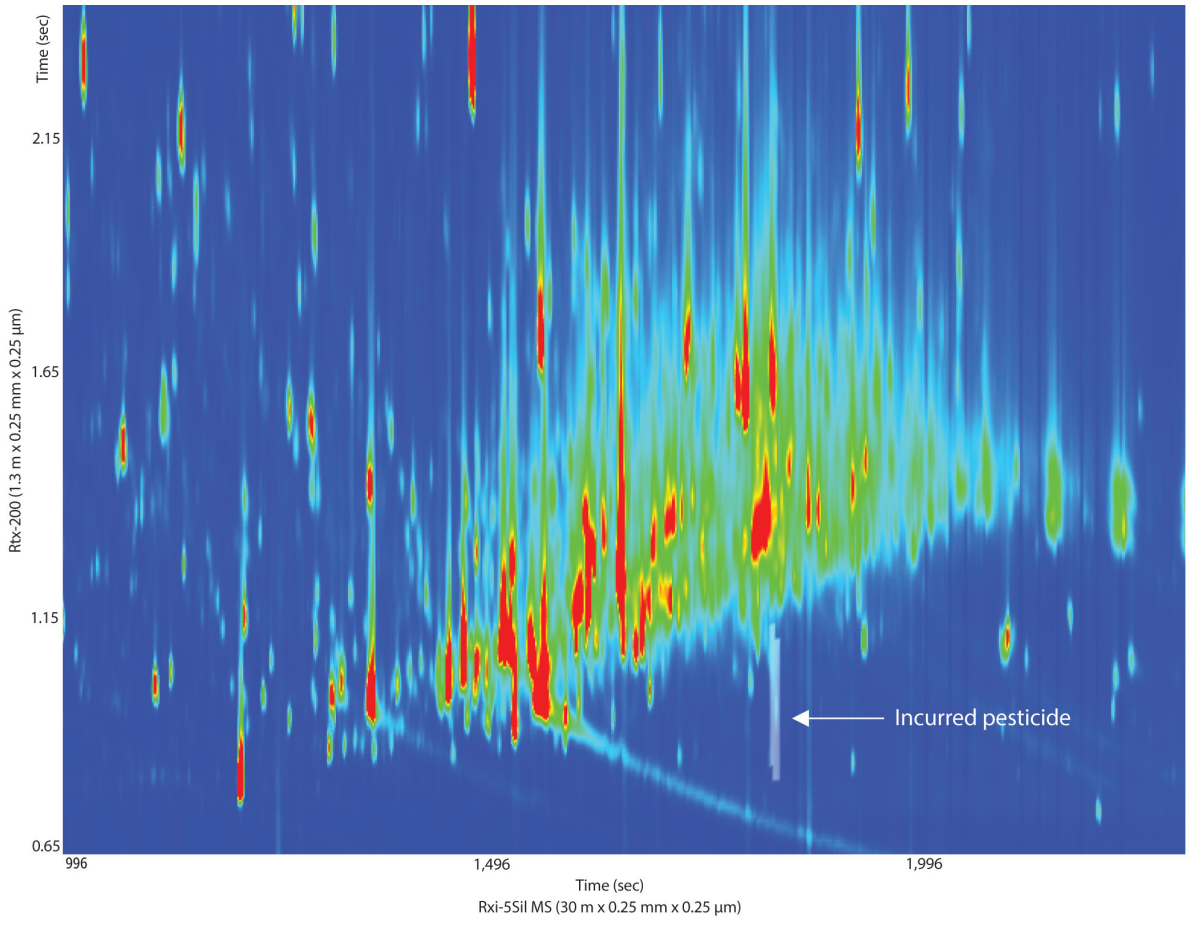
GCxGC-TOFMS Analysis of Unfortified Tobacco Extract With Incurred Pesticide



GC_FF1243

Column	Rxi-5Sil MS 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623) Rtx-200 1.3 m, 0.25 mm ID, 0.25 µm (cat.# 15124)
Standard/Sample	Tobacco extract with dSPE cleanup
Injection	
Inj. Vol.:	1.0 µL splitless (hold 1.0 min)
Liner:	Premium 4 mm single taper w/wool (cat.# 23303)
Inj. Temp.:	250 °C
Oven	
Oven Temp.:	Rxi-5Sil MS: 90 °C (hold 1.0 min) to 310 °C at 5 °C/min (hold 2.0 min) Rtx-200: 95 °C (hold 1.0 min) to 315 °C at 5 °C/min (hold 2.0 min)
Carrier Gas	He, corrected constant flow (2 mL/min)
Modulation	
Modulator Temp. Offset:	+20 °C
Second Dimension Separation Time:	3 sec
Hot Pulse Time:	0.9 sec
Cool Time between Stages:	0.6 sec
Detector	MS
Mode:	
Transfer Line Temp.:	300 °C
Analyzer Type:	TOF
Source Temp.:	225 °C
Electron Energy:	70 eV
Mass Defect:	-20 mu/100 u
Ionization Mode:	EI
Acquisition Range:	45 to 550 amu
Spectral Acquisition Rate:	100 spectra/sec
Instrument	LECO Pegasus 4D GCxGC-TOFMS
Sample Preparation	*QuEChERS Extraction:* A 2 g sample of tobacco was weighed into a 50 mL polypropylene centrifuge tube (cat.# 26239). After the addition of 10 mL of organic-free water to the sample, 100 µL of QuEChERS internal standard mix for GC-MS analysis (cat.# 33267) was added to each sample. Next, 10 mL of acetonitrile was added and the samples were vortexed for 30 min. Immediately after vortexing, pre-packaged QuEChERS European EN 15662 method formulation extraction salts (cat.# 26236) were added to each centrifuge tube. The tubes were immediately shaken for 1 min and then centrifuged for 5 min at 3000 g. *Sample Cleanup:* A 1 mL aliquot of the extract was fortified with 5 µL of an anthracene standard (cat.# 33264) and added to a dSPE tube containing 150 mg $MgSO_4$, 25 mg PSA and 7.5 mg GCB (cat.# 26218). The tubes were gently shaken for 2 min and then centrifuged for 5 min using a Q-sep 3000 centrifuge (cat.# 26230). A 0.5 mL portion of the supernatant extract was removed and placed into an autosampler vial and 5 µL of a 5% formic acid solution in acetonitrile was added to each sample prior to analysis.
Notes	Rtx-200 (cat.# 15124) is a 2 m column. A 1.3 m section was cut off and used as the second dimension column.