Pretreatment

1. Add 15 g of ground potatoes into a 50 mL centrifuge tube.
2. Add 15 mL 1% acetic acid in acetonitrile with 1.5 g anhydrous sodium acetate and 6 g anhydrous magnesium sulfate.
3. Shake vigorously for 1 minute.
4. Centrifuge >1500 rcf for 1 minute.
5. Take out 7.5 mL extract and dilute to 10 mL with 2.5 mL toluene.

SPE Procedure

Sep-Pak® Vac Carbon Black/Aminopropyl 6 cc/500 mg/500 mg

CONDITION:
10 mL 25:75 toluene: actonitrile (v/v)

↓

Add 200 mg anhydrous magnesium sulfate to top of cartridge to remove water

↓

LOAD:
Extract (collect)

↓

ELUTE:
10 mL 25:75 toluene: acetonitrile (collect)

↓

Combine both collected fractions and adjust volume to exactly 20 mL by addition of toluene:acetonitrile (25:75, v/v)

↓

Take 5 mL and evaporate to just below 1 mL and bring up to 1 mL with toluene. Inject onto GC/MS.

GC Conditions

Instrument:	Agilent 6890
GC column:	DB-5ms, 30m x 0.25mm (ID), 0.25 µm film (Agilent). Direct connection of column to injection-port liner
Tranfer line to MS:	300 °C
Source temperature:	200 °C
Injection volume:	1 µL splitless
Injection port temperature:	180 °C
Initial temperature:	80 °C
Time at initial temperature:	1 minute

Then program at 10 °C/ min to 200 °C

Then at 25 °C minute to 300, hold 5 minutes

GC/MS Conditions

Instrument:	Waters Quattro micro™ GC
Ionization mode:	Positive electrospray (70 eV) Selected-Ion Recording (SIR)

HP6890 GC Flow 1

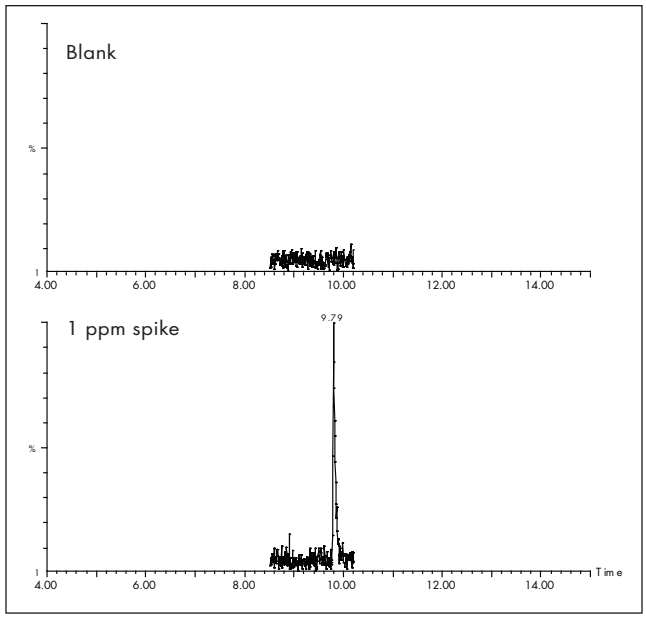
Initial Flow: 1 mL/min

Time (min)	Rate (mL/min)	Final Flow (mL/min)
0	50	3
0.5	50	3
0.6	50	1

GC-MS (SIR)

Channel	Mass
1 (Quantification)	92.8
2 (Confirmation)	119
3 (Confirmation)	120

Results



Chromatogram of a 1 µg/g spiked potato sample.

Compound Name: propham 92.8	RT	Area
1 ppm spiked 1	9.78	379.00
1 ppm spiked 2	9.82	382.00
1 ppm spiked 3	9.80	458.00
1 ppm spiked 4	9.79	399.00
1 ppm spiked 5	9.75	421.00
Mean		407.80
RSD (%)		8.01
Recovery (%)		95.73

Recovery results for 1 µg/g spiked potato sample.

Ordering Information

Description	Part Number
Sep-Pak Vac Carbon Black/Aminopropyl, 6 cc/500 mg/500 mg	186003369
LCMS Certified Vials	600000751CV