

Pretreatment

1. Extract a 20 g sample with 50 mL of acetonitrile.
2. Filter extract. Collect filtrate and bring to 100 mL final volume with acetonitrile.
3. Add 10 g of sodium chloride and 20 mL of 0.5 M phosphate buffer (pH 7) to 20 mL of diluted extract.
4. Shake and allow the liquid layers to separate. Remove aqueous layer and discard.
5. Collect the acetonitrile fraction and add sodium sulphate to dry the solvent.
6. Evaporate and reconstitute in 2 mL of 3:1 acetonitrile:toluene.

SPE Procedure

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

CONDITION: Toluene/acetonitrile 10 mL (1:3 v/v). Do not allow to dry
LOAD: Rinse vials with toluene/acetonitrile (1:3 v/v) solution and load 2 mL of extracted sample
ELUTE : 20 mL toluene/acetonitrile (1:3 v/v). Flow: 2 mL/min

LC Conditions

Instrument: Waters Alliance® HPLC 2695 System
 Column: Xterra® MS C₁₈, 2.1 x 150 mm, 3.5 µm
 Flow rate: 0.2 mL/min
 Mobile phase:
 A. water
 B. methanol
 C. 100 mM ammonium acetate in water

Gradient: Time (min) A% B% C%
 0 80 15 5
 1 55 40 5
 3.5 55 40 5
 6 45 50 5
 8 40 55 5
 17.5 0 95 5
 30 80 15 5
 47 80 15 5

Injection volume: 5 µL
 Temperature: 40 °C

MS Conditions

Instrument: Waters Quattro Premier™ XE
 Ionization mode: Positive electrospray (ESI⁺)
 Multiple reaction monitoring

Results

Pesticides*	Spike Conc. / µg/g	Recovery (%)
Abamectin	0.1	102.0
Anibfos	0.1	111.7
Azinphos-methyl	0.1	107.6
Benzofenap	0.1	139.5
Butafenacil	0.1	104.5
Chloridazon	0.1	106.0
Chromafenozone	0.1	108.2
Clomeprop	0.1	104.4
Cloquintocet-mexyl	0.1	108.7
Clothianidin	0.1	101.5
Cyazofamid	0.1	108.3
Cyflufenamid	0.1	110.1
Dimethirimol	0.1	101.0
Fenoxycarb	0.1	108.7
Ferimzone	0.1	112.6
Formetanate hydrochloride	0.1	86.7
Furathiocarb	0.1	100.5
Imidacloprid	0.1	111.8
Indoxacarb	0.1	121.2
Iprovalicarb	0.1	106.2
Isoxaflutole	0.1	99.5
Lactofen	0.1	106.8
Methoxyfenozide	0.1	103.3
Mibemectin A3	0.1	114.5
Mibemectin A4	0.1	101.2
Naproanilide	0.1	115.9
Oryzalin	0.1	103.8
Oxycarboxin	0.1	85.1
Oxydemeton-methyl	0.1	108.0
Phenmedipham	0.1	102.2
Pyrazolynate	0.1	72.7
Quizalofop-P-tefuryl	0.1	145.3
Simeconazole	0.1	106.0
Thiacloprid	0.1	109.2
Thiamethoxam	0.1	108.3
Tridemorph	0.1	94.6
Etriconazole	0.1	113.3

*Five replicate samples analyzed per level.

Ordering Information

Description	Part Number
Sep-Pak Vac Carbon Black/Aminopropyl, 6 cc/500 mg/500 mg	186003369
Xterra MS C ₁₈ , 2.1 x 150 mm, 3.5 µm	186000506
LCGC Certified Vials	186000272C