

Determination of Pesticides

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This is a method for the analysis of pesticide residues in foods of plant origin, such as fruits (including dried fruits), vegetables, cereals and processed products thereof. The method can analyze 300 components at the same time, using acetonitrile extraction/partitioning and SPE – QuEChERS method for clean-up followed by GC-MS and/or LC-MS/MS. In this example, only 10 components were determined though.

Reagents:

(only use recognized analytical grades, unless otherwise specified)

Water (1.15333)
Acetonitrile (1.00029)
Methanol, LC-MS grade
Ammonium formate, LC-MS grade
Formic acid (1.00264.1000)
Magnesium sulfate anhydrous (1.06067)
Sodium chloride (1.06404)
Bondesil PSA, 40um, 100 g
Carbon SPE Bulk Sorbent, 25g bottle

Pesticide Standards

Azoxystrobin
Buprofezin
Fenpyroximate
Hexythiazox
Myclobutanil
Penconazole
Tetraconazole
Tolyfluanid
Trifloxystrobin
Triflumizole

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Sample preparation (tomatoes)

1. Take a representative sample (10 g) a place in a suitable container
2. Add 10 ml of formic acid: acetonitrile (1:1 volume/volume-%) and homogenize
3. Add buffer-salt mixture, homogenize
 - Magnesium sulfate (4 g)
 - Sodium chloride (1 g)
 - Centrifuge 4200 rpm, 2.5 min
4. Take as much as possible from the upper phase (4 mL)
5. Add sorption mixture salt to this phase, homogenize
 - Carbon SPE Bulk Sorbent (35.0 mg)
 - Bondesil-PSA (113.0 mg)
 - Magnesium sulfate (652.0 mg)
 - Centrifuge 4200 rpm, 2.5 min
6. Fill 1 ml from the upper phase into a vial

LC-MS/MS analysis (use an appropriate UHPLC system)

MS-MS detector: Q Trap MS/MS system or similar

HPLC column: Fused core C18 (10 cm x 3,0 mm, 2,7µm)

Guard Column: Fused core C18 (0.5 cm x 3,0 mm, 2,7µm)

Eluent system: A: 1 mmol/l ammonium formate with 0.1% formic acid in water

B: Methanol

Flow rate: 500 µL/min

Injection: 20 µL

Temperature: 40 C°

Gradient profile:

Time (min)	A (%)	B (%)
0	95	5
2.0	65	35
8.5	5	95
15.0	5	95
16.0	95	5
20.0	95	5

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MS-MS parameters	
Polarity	+
CUR	40 psi
CAD	High
IS	5500 V
Temp	400 °C
GS1	35 psi
GS2	45 psi

	Q1	Q3	DP-(V)	EP- (V)	CE- (V)	CXP-(V)	RT (min)
Azoxystrobin 1	404.1	372.3	31	6.5	19	6	11.04
Azoxystrobin 2	404.1	344.2	31	6.5	25	6	11.04
Buprofezin 1	306.1	201.3	50	4	17	4	13.34
Buprofezin 2	306.1	106.0	50	4	31	4	13.34
Fenpyroximate 1	422.3	366.3	46	6	21	4	14.62
Fenpyroximate 2	422.3	135.2	46	6	45	4	14.62
Hexythiazox 1	353.3	228.0	41	6.5	19	4	14.02
Hexythiazox 2	353.3	168.3	41	6.5	31	4	14.02
Myclobutanil 1	289.1	70.2	41	5.5	31	4	11.56
Myclobutanil 2	289.1	125.1	41	5.5	39	4	11.56
Penconazole 1	284.2	159.1	36	5.5	35	4	12.28
Penconazole 2	284.2	70.2	36	5.5	29	4	12.28
Tetraconazole 1	372.1	159.0	46	4	39	4	11.79
Tetraconazole 2	372.1	70.1	46	4	39	4	11.79
Tolyfluanid 1	347.2	137.2	41	5.5	37	4	12.23
Tolyfluanid 2	347.2	238.0	41	5.5	15	4	12.23
Trifloxystrobin 1	409.3	186.1	41	4	23	4	12.85
Trifloxystrobin 2	409.3	206.3	41	4	19	4	12.85
Triflumizole 1	346.2	278.3	36	4	15	4	12.89
Triflumizole 2	346.2	73.1	36	4	23	4	12.89

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