



Analysis of Amitraz and its Main Metabolite in Pears via QuEChERS and LC-MS/MS

- Brief Description -

Standards:

Amitraz: for example from Dr Ehrenstorfer GmbH, Germany; product no. C10230000, certified

N-2,4-dimethylphenyl-N-methylformamidine: for example from Dr Ehrenstorfer GmbH, Germany; product no. L 12738000AC, 10 ng/μl

Internal standard triphenyl phosphate: for example from Aldrich; >99%; product no. 241288

QuEChERS extraction:

- Weigh 10 g of the comminuted, homogeneous, frozen sample into a 50 ml centrifugation tube
- Add 10 ml acetonitrile and 100 μl internal standard solution (c = 20 μg/ml); close the tube and shake vigorously for 1 min.
- Add 4 g Magnesium sulphate anhydrous, 1 g sodium chloride, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate and shake for 1 min; Centrifuge for 5 min at 3000 rpm
- Transfer an aliquot of the extract into a centrifugation tube which contains 25 mg PSA and 150 mg magnesium sulphate per mL extract; shake the tube vigorously for 30 s and centrifuge for 5 min at 3000 rpm.
- Fill an aliquot of the extract into a vial and employ for LC-MS/MS analysis

Analysis via LC-MS/MS (Please consider: the LC-MS/MS data is exemplary.)

Instrument: API 4000

Mode: ESI positive

MRMs: amitraz: 294/121; 294/163; N-2,4-dimethylphenyl-N-methylformamidine: 163/107; 163/132

Column: Zorbax XDB Eclipse 150 x 2 mm; 3.5μ

Solvent: A: 5 mmol NH₄-formiate in water; B: 5 mmol NH₄-formiate in methanol

Flow: 0,3 ml/min

Injection volume: 5 μl

Recovery: 84 - 115% (for details see www.crl-pesticides-datapool.eu;

→ Pesticide: Dimethylphenyl-N-methylformamidine, N-2,4-)

LOQ: amitraz: 0,005 mg/kg; N-2,4-dimethylphenyl-N-methylformamidine: 0,005 mg/kg

Community Reference Laboratory for Single Residue Methods

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