



Modified Version of the QuEChERS-Method for the Analysis of Pentachlorophenol in Guar Gum - Brief Description -

Extraction

- Weigh 1 g of homogenous sample into a 50 mL centrifuge tube
- Add 10 mL of acetonitrile and shake for a few seconds
- Add 100 μ L of internal standard solution (Nicarbazin) and shake for a few seconds
- Add 10 mL of water
- Shake vigorously for 1 min
- Add 4 g MgSO_4 , 1 g NaCl, 1 g Na_3 -citrate dihydrate and 0.5 g Na_2H -citrate - sesquihydrate
- Shake for 1 min
- Centrifuge for 5 min at 3000 rpm
- Fill an aliquot of the extract (0.1 g/IS) into a vial and employ for analysis.

Analysis by LC-MS/MS

Instrument: for example API 3200

Mode: ESI negativ

PCP-MRMs: 263/35, 265/35 and 267/35

Column: Zorbax XDB Eclipse 150x2mm 3.5 μ

Solvent: A: 5 mmol NH_4 formiate in water, B: 5 mmol NH_4 formiate in methanol

Flow: 0.2 mL/min

Gradient: 10 % B \rightarrow 90 % B: in 8 min, 90 % B: 8 min - 15 min, temp. 40 $^{\circ}\text{C}$

Injection Volume: 5 μ L

Recovery: 70 % - 110 % (Detailed validation data can be found at

<http://www.crl-pesticides-datapool.eu/>)

LOD: 0.01 mg/kg