Analysis of Organotin Compounds via QuEChERS and LC-MS/MS

- Brief Description -

Sample extraction (QuEChERS):

Detailed guidelines for the sample preparation (QuEChERS method) are given in the CEN method EN 15662 (Determination of pesticide residues using GC-MS and/or LC-MS (/MS) following acetonitrile extraction/partitioning and cleanup by dispersive SPE - QuEChERS method) or as brief description on the Internet (www.quechers.de).

Analysis by LC-MS/MS:

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

Analyte scope	Cyhexatin (sum of azocylotin and cyhexatin) Fentin Fenbutatin oxide Internal Standard: TPP
Instrument parameters	API 4000
Ionisation mode	ESI pos
Column	Zorbax 3,5 µm; Eclipse XDB-C18; 2,1x 50 mm
Pre-column	C18 ODS 4mm x 2mm ID (Phenomenex AJO-4286)
Eluent A	5 mmol NH_4formiate in H_2O +1% formic acid
Eluent B	5 mmol NH_4formiate in Methanol +1% formic acid
Gradient	flow: 0.4ml/min time A B 0 60% to 40% 2 0% to 100% 8 0% to 100% 8.1 60% to 40% 13 60% to 40%

Aquired mass transitions

Analyte, mass transition (m/z)	Relative Sensitivity 6= best)	First Mass	Second Mass		DP	CE	CXP
Cyhexatin 365/201	**5**	**365.1**	**200.9**		**71**	**27**	**30**
Cyhexatin 365/283	**6**	**365.1**	**283.1**		**71**	**17**	**8**
Cyhexatin 367/203	**3**	**367.2**	**202.9**		**71**	**25**	**28**
Cyhexatin 367/285	**4**	**367.2**	**285.0**		**71**	**17**	**10**
Cyhexatin 369/205	**1**	**369.2**	**205.0**		**71**	**23**	**18**
Cyhexatin 369/287	**2**	**369.2**	**287.0**		**71**	**15**	**18**
Fentin 347/116	**6**	**347.0**	**115.9**		**96**	**43**	**20**
Fentin 347/193	**5**	**347.0**	**193.0**		**96**	**37**	**16**
Fentin 349/118	**4**	**349.0**	**117.9**		**91**	**41**	**20**
Fentin 349/195	**3**	**349.0**	**195.0**		**91**	**37**	**16**
Fentin 351/120	**2**	**351.0**	**119.9**		**96**	**41**	**8**
Fentin 351/197	**1**	**351.0**	**197.1**		**96**	**37**	**14**
Fenbutatin oxide 517/195	**6**	**517.3**	**195.0**		**116**	**79**	**18**
Fenbutatin oxide 517/349	**5**	**517.3**	**349.0**		**116**	**45**	**12**
Fenbutatin oxide 517/461	**4**	**517.3**	**461.1**		**116**	**35**	**14**
Fenbutatin oxide 519/197	**2**	**519.3**	**197.0**		**121**	**67**	**16**
Fenbutatin oxide 519/351	**3**	**519.3**	**350.9**		**121**	**47**	**20**
Fenbutatin oxide 519/463	**1**	**519.3**	**463.3**		**121**	**35**	**14**

Quantification:

The analyte concentration is calculated via regression curve of the area ratios of the analyte to the internal standard. In case of suspected MRL-violations a standard addition procedure is used.

Performance:

Reporting levels using API 4000 instrumentation: 0.002 mg/kg for all compounds

Validation data CVUA Stuttgart brief overview:

Pesticide	Level min	Level max	Rec Median	Rec Mean	CV [%]	# of rec	# of outl	% Rec (70-120%)	# of Labs
Cyhexatin	0,1	0,1	**83**	84	11,9	20	0	**90**	1
Fenbutatin Oxide	0,04	0,1	**93**	91	13,3	21	0	**95**	1
Fentin	0,1	0,1	**86**	86	9	20	0	**100**	1

For more data see **www.crl-pesticides-datapool.eu**, method validation data