EU PROFICIENCY TEST
EUPT-SRM10, 2015

Residues of Pesticides requiring Single Residue Methods

Test Item:

Maize Flour

Final Report

Supplementary Information on Analytical Methods

S-1 Methods used by the participating laboratories (ordered by Lab-Codes)

Compulsory analytes S-2

Optional analytes S-74

S-2 Methods used by the participating laboratories (ordered by z-scores)

Compulsory analytes S-100

Optional analytes S-172

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | 2,4-D (free acid)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		Yes	> 2 y	0.095	0.1	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	97 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.082	-0.4	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML	$MCPP-D_3$	PrCal	112 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.098	0.3	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	115 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	1 - 2 y	0.127	1.5	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	$2,4-D\ D_3$	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.095	0.1	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	2.5-dichloroben-zoic acid	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.098	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.09	-0.1	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	86 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM
9		Yes	1 - 2 y	0.092	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	20 % NaOH; pH 12	Isooctane	1× (3-4)			SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	83 % (not cor-rected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
10		Yes	> 2 y	0.106	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	$MCPA\ D_6$	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		Yes	> 2 y	0.096	0.2	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		101 % (0.02 and 0.1 mg/kg)	SB-EUPT	3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
12	x	Yes	1 - 2 y	0.098	0.3	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	100.4 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
15		Yes	> 2 y	0.0923	0.0	0.004	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	95 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
16		Yes	> 2 y	0.065	-1.2	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	63.5 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
18		Yes	> 2 y	0.068	-1.0	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.11	0.8	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93.5 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0767	-0.7	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	71 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		Yes	> 2 y	0.088	-0.2	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
22		Yes	> 2 y	0.103	0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4)		No	Disp.-SPE ($PSA/MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	97 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
23	x	Yes	1 - 2 y	0.118	1.1	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE ($PSA/MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	110 %	SB-EUPT	3	other

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
24	x	No	> 2 y	0.0131	-3.4	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
25		Yes	> 2 y	0.106	0.6	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	95 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
26	x	No	> 2 y	0.092	0.0		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	64.7 %		3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.111	0.8	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	107 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
28		No	> 2 y	0.078	-0.6	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-dimethyl-phe-noxy) HOAc	None	90 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.094	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	103 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
30		Yes	> 2 y	0.105	0.6	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabu-tyl-ammoni-umhydroxide/ iodomethane		GC-MSD (following derivatization), m/z: 401/400/391	PS-SL	No	None	88 %	SB-EUPT	1	alkaline hydrolysis extraction, GPC, acid/base distribution, methylation, GC-MSD detection
31		Yes	< 1 y	0.062	-1.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	120 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
32		Yes	> 2 y	0.082	-0.4	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.135	1.9	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
34		Yes	> 2 y	0.026	-2.9	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Centrifugation	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	76 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
35		Yes	> 2 y	0.0706	-0.9	0.01	5	ambient	10 ml	No	mechanical shaking, 30 min	No	EtOAc with 1 % HOAc	1× (1 % HOAc)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	72 % (0.101 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time
36		Yes	> 2 y	0.109	0.7	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	RecF	61 % (Avegrage from ongoing AQC samples)	QC	> 5	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
37	x	Yes	> 2 y	0.095	0.1	0.05	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	108 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA cleaning
38		Yes	> 2 y	0.083	-0.4	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
40		Yes	> 2 y	0.092	0.0	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifugation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	80 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.081	-0.5	0.005	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA				QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	> 2 y	0.1	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)

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45		Yes	> 2 y	0.0986	0.3	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	92 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
46		Yes	1 - 2 y	0.108	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	95 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
47	x	No	None	0.123	1.3	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	81.8 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.088	-0.2	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	StAdd-EA	nicarbazin	StAdd-EA	101 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.1	0.3	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	122 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.092	0.0	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	99 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 with extract conc
55		Yes	> 2 y	0.092	0.0	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	(3-chloro-4-methyl-phenoxy)-HOAc	PrCal	129 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
56		Yes	> 2 y	0.0854	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	98 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.092	0.0	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	113 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
58		Yes	> 2 y	0.049	-1.9	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
59	x	Yes	> 2 y	0.072	-0.9	0.05	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min	NaOH	ACN	1× (H_2SO_4)		Yes	C18/$MgSO_4$	GC-MSD (following derivatization)	MM-ML	No	None	77 %	SB-EUPT	2	other (with derivatization)
60	x	No	> 2 y	0.0829	-0.4	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	103 %	SB-EUPT	3	Modified QuEChERS
61		Yes	> 2 y	0.09	-0.1	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	96 %	QC		QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.13	1.6	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	55 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
64	x	Yes	> 2 y	0.083	-0.4		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	other	28.9 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step; Sample corrected for QC recovery
65	x	Yes	> 2 y	0.0735	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	70 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	> 2 y	0.1	0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.1106	0.8	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		95 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
70	x	Yes	> 2 y	0.0906	-0.1	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	62 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
71		No	None	0.133	1.8	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87.4 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)

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- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
72		Yes	> 2 y	0.0982	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
73		No	None	0.0849	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	93.5 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.076	-0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
75		Yes	> 2 y	0.104	0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA	84 %	QC	> 5	A-QuEChERS (with 1 % FA)
76		Yes	> 2 y	0.08	-0.5	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No				LC-Orbitrap	PS-ML	2.4,6-trichloro-phenol	None	93 %			A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.086	-0.3	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
78		Yes	> 2 y	0.058	-1.5	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifuga-tion	LC-MS/MS (QQQ)	PS-ML	generic IS	None	62 %	SB-EUPT	2	in house method
79		No	< 1 y	0.055	-1.6	0.05	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
80		Yes	> 2 y	0.105	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.084	-0.4	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
82		Yes	> 2 y	0.112	0.9	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	None			4	QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.086	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
84	x	Yes	> 2 y	0.087	-0.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
85		No	> 2 y	0.076	-0.7	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		62 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
87		Yes	> 2 y	0.095	0.1	0.02	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	67.3 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
89		Yes	< 1 y	0.104	0.5	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	76 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
90		Yes	> 2 y	0.0875	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	91 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
91	x	Yes	> 2 y	0.0826	-0.4	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.4 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
94	x	Yes	> 2 y	0.111	0.8	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	105.9 % (0.01 mg/Kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | 2,4-D (free acid)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
97	x	Yes	> 2 y	0.06	-1.4	0.02	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/$MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	nicarbazin	None	75 % (0.02 mg/kg)	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM, EURL SRM
98	x	Yes	1 - 2 y	0.102	0.4	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		91 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.14365	2.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	122.19 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.093	0.0	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	69 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
106		Yes	> 2 y	0.11	0.8	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	91 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
107		Yes	> 2 y	0.082	-0.4	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	0.125	1.4	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	110 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
114		No		0.028	-2.8	0.01														other compound	StAdd-SP	104 %	SB-EUPT	1	no data
115		Yes	None	0.03	-2.7	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
116		Yes	> 2 y	0.11	0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
117		Yes	1 - 2 y	0.104	0.5	0.01	2	just thawed	Yes	H_2O,	manual shaking		ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	91 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
118		No	None	0.0853	-0.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Chlormequat

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	> 2 y	0.208	1.0	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	31 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
2		Yes	> 2 y	0.155	-0.3	0.01	10	deep frozen	No	No	mechanical shaking, 60 min	No	H_2O / MeOH / HCl	No		No	Disp.-SPE; alumina	LC-MS/MS (QQQ)	PS-ML	chlormequat-D_4	None	91 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.137	-0.7	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.159	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	chlormequat-D_4	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.200	0.8	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	Yes	> 2 y	0.136	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
8		No	> 2 y	0.226	1.4	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		Isooctane				No	LC-MS/MS (QQQ)		ILIS	other	93 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
9		Yes	> 2 y	0.167	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				Filtration	LC-MS/MS (QQQ)	PS-ML	isotope D_4	PrCal	103 % (not corrected)	SB-EUPT	3	SIST EN 15055:2006
10		Yes	> 2 y	0.212	1.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	None	96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.159	-0.2	0.01	10	deep frozen	10 ml	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ)	MM-ML	D_4-chlorme-quatchlorid		96 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	other
12	x	Yes	> 2 y	0.168	0.0	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	StAdd-EA	98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.152	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	81 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.158	-0.2	0.004	5	ambient	No	after H_2O and organic solvent, 10 min	mechanical shaking, 20 min	No		1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	84 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), extraction mixture: 20 mL 80 % ACN in H_2O, 1 % FA
16		Yes	< 1 y	0.136	-0.8	0.01	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	chlormequat chloride D_4	None	82.4 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
18		Yes	> 2 y	0.154	-0.3	0.01	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
19		Yes	1 - 2 y	0.13	-0.9	0.05	5	ambient	No	No	5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	88.6 % (0.050 and 0.50 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	> 2 y	0.172	0.1	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		chlormequat chloride D_4	StAdd-SP	74 %	SB-EUPT	2	MeOH extraction
21		Yes	> 2 y	0.171	0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	89 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
23	x	Yes	1 - 2 y	0.171	0.1	0.01	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	101 %	SB-EUPT	3	other (EN 15055, 2006-08)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
25		Yes	> 2 y	0.214	1.1	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	PrCal	102 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
26	x	No	> 2 y	0.183	0.4		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	85.4 %		3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	> 2 y	0.17	0.1	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	104 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
29	x	Yes	> 2 y	0.154	-0.3	0.01	5	slightly frozen	No		mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		No	Centrifugation, Filtration, nylon filter	LC-MS/MS (QQQ), Waters	MM-ML	chlormequat-D_4	None	91 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.179	0.3	0.005	5	ambient	19.5 mL	after H_2O and organic solvent, 5 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ), 122/58	PS-ML	D_4-chlormequat	None	93 % (84 % D_4-CCC recovery)	SB-EUPT	1	DIN EN 15055, 2006-08
31		Yes	> 2 y	0.28	2.7	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	114 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
32		No	> 2 y	0.24	1.7	0.01	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.154	-0.3	0.01	5	ambient	5 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	chlormequat-D_4					in house method
34		Yes	> 2 y	0.12	-1.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	80 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
35		Yes	> 2 y	0.161	-0.2	0.005	10	ambient	20 ml	No	mechanical shaking, 15 min	No	Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	chlormequat chloride D_4	None	95 %	SB-EUPT	1	Startin J.R., Hird S.J., Sykes M.D., Taylor J.C. and Hill A.J. Determination of residues...
36		Yes	> 2 y	0.0804	-2.1	0.01	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_4 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.162	-0.1	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS	MM-ML	No	StAdd-SP	61.1 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.134	-0.8	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides); Dilution of extract 1:50
43		Yes	> 2 y	0.155	-0.3	0.008	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	PS-ML	D_4-chlormequat					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
44	x	Yes	> 2 y	0.198	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		Yes	> 2 y	0.15	-0.4	0.005	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	91 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.178	0.3	0.005	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	None	99 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
47	x	Yes	> 2 y	0.168	0.0	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	Isooctane				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	78.8 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
49	x	No	> 2 y	1.35	28.3	0.01	5	cold	Yes	after H_2O, 5 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ILIS	None	100 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM
51	x	Yes	> 2 y	0.169	0.0	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		109 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
54	x	Yes	> 2 y	0.153	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, Ammonium Acetate	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	StAdd-SP	No	StAdd-SP	69 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 without dilution
55		Yes	> 2 y	0.258	2.2	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	93 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
56		Yes	1 - 2 y	0.156	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/$MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	25 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		No	1 - 2 y	0.117	-1.2	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	> 2 y	0.194	0.6	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
59	x	No	< 1 y	0.163	-0.1	0.02	10	cold	20 ml	after H_2O, 10 min	manual shaking, 2 min	No	Isooctane	No		No	Centrifugation	LC-Ion Trap	PS-ML	ILIS		110 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.166	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Disp.-SPE (ODS/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	97 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.15	-0.4	0.01	5	cold	No	No	mechanical shaking, 10 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	98 %	QC		Shaking with MeOH
63		Yes	< 1 y	0.194	0.6	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN	Yes (HOAc buffered)			Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	50 %	SB-EUPT		QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
64	x	No	> 2 y	0.168	0.0	0.02	5	deep frozen	10 ml	after H_2O, 10 min		No	H_2O/HCl, Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95.1 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.249	1.9	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	75 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
66		Yes	> 2 y	0.185	0.4	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	102 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
67	x	Yes	> 2 y	0.1542	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.167	0.0	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	102 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	Yes	> 2 y	0.17	0.1	0.01	25	cold	Yes	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	D_4-chlormequat	None				MeOH/H_2O extraction
75		Yes	> 2 y	0.17	0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	chlormequat-D_4	StAdd-EA		QC	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
77		No		0.245	1.9	0.01												no data		No	StAdd-SP				no data
78		Yes	> 2 y	0.1	-1.6	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	68 %	SB-EUPT	1	in house method
80		Yes	> 2 y	0.197	0.7	0.01	10	ambient	10 ml	after H_2O and organic solvent, 10 min	ultra turrax, 1 min	No	ACN	No		No	SPE-column; OASIS WCA	LC-MS/MS (QQQ), Waters Xevo	StAdd-SP	chlormequat-D_4	StAdd-SP				other
81	x	Yes	> 2 y	0.171	0.1	0.01	10	cold	19 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	chlormequat chloride D_4	None	107 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.167	0.0	0.01	5	ambient	10 ml	No	ultrasonic bath, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
83		Yes	> 2 y	0.158	-0.2	0.01	10	ambient			mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), using 120 ml of extraction solution
84	x	Yes	> 2 y	0.163	-0.1	0.01	10	slightly frozen	Yes	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	137 %	SB-EUPT	1	EN15055
85		No	< 1 y	0.065	-2.4	0.01	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		67.2 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		Yes	> 2 y	0.172	0.1	0.015	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Centrifugation	LC-MS/MS (QQQ), ESI pos.	MM-ML	$CCC-D_4$	None	84.9 %	SB-EUPT	1	§ 64 LFGB, L00.00-76
89		Yes	< 1 y	0.197	0.7	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min		Isooctane	1× (FA)				LC-MS/MS (QQQ)	MM-ML	TPP	PrCal	120 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
90		Yes	> 2 y	0.165	-0.1	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane, acidified with FA	1× (by FA within the solvent)		No	Filtration; 0.45 µm	LC-MS/MS (QQQ)	MM-ML	No	None	85 % (0.08 mg/kg)	SB-EUPT	1	§ 64 LFGB, L00.00-76
91	x	No	> 2 y	0.183	0.4	0.01	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	1 % FA in ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	No	None	95.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), solvent = 1 % FA in ACN, no ISTD was used
94	x	Yes	> 2 y	0.166	0.0	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93.4 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.18	0.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
97	x	No	> 2 y	0.09	-1.8	0.05	10	ambient	until volume to 20 mL	No	mechanical shaking, 2 min	No	Isooctane, 40 mL MeOH	No		No	No	LC-MS/MS (QQQ), internal standard	MM-SL	$chlormequat-D_4$	None	62 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), analysis of chlormequat and mepiquat residue in foods of plant origin EURLSRM Jan 2009
98	x	Yes	1 - 2 y	0.172	0.1	0.01	5	deep frozen	Yes	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Centrifugation	LC-MS/MS (QQQ)	MM-ML	No		62 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.6009	10.4	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	146.64 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
106		Yes	> 2 y	0.21	1.0	0.01	10	deep frozen	Yes		15 min	No						LC-MS/MS (QQQ)	MM-ML	ILIS	None	91 %	SB-EUPT	1	other (extraction with MeOH)
107		Yes	> 2 y	0.0614	-2.5	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	None	79 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
111		Yes	> 2 y	0.162	-0.1	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	$chlormequat-D_4$	None	93 % (2x MRRL)	SB-EUPT	1	other
113		Yes	> 2 y	0.192	0.6	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	$chlormequat-D_4$	None	90 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
115		Yes	None	0.02	-3.5	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
116		Yes	> 2 y	0.12	-1.1	0.01	2	ambient	Yes	after H_2O and organic solvent, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
118		No	None	0.00094	-4.0		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-SL	$chlormequat-D_4$	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavge step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	> 2 y	0.59	0.2	0.003	1	deep frozen	4 ml	No		$SnCl_2$/HCl, 15 min	H_2O/HCl	HCl		No		GC-MSD, Headspace	StAdd-SP	DCM	StAdd-SP	79 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
2		Yes	> 2 y	0.635	0.5	0.05	80	deep frozen	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No					$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
3		Yes	> 2 y	0.698	1.0	0.02	2	cold	2 ml	after H_2O, min	mechanical shaking, 45 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-Ion Trap	MM-ML	No	None	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
4	x	No	None	0.95	2.8	0.05	50	slightly frozen	45 mL	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, 2h	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	No	None	55 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
5		Yes	> 2 y	0.32	-1.7	0.05	5	ambient	No	No		$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	StAdd-SP	thiophene	PrCal	95 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
6	x	Yes	> 2 y	0.458	-0.7	0.04	25	ambient				$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	MM-ML	DCM	PrCal	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
7		Yes	> 2 y	0.532	-0.2	0.05	50	ambient	No	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	No	None	70.2 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
8		No	> 2 y	0.45	-0.8	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	manual shaking	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MS/MS (QQQ)		No	other	86 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
9		Yes	> 2 y	0.774	1.5	0.05	25	cold	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	85 % (not corrected)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
10		Yes	> 2 y	0.462	-0.7	0.05	25	ambient	25 ml	after H_2O and organic solvent, 5 min	mechanical shaking, > 60 min	$SnCl_2$/HCl, release of CS_2	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)		No	No	GC-MSD	PS-ML	No	None	89 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, GC-MS detection
11		Yes	> 2 y	0.547	-0.1	0.05	20	deep frozen				$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No	None	97 % (CS_2 = 0.253 mg/kg from Thiram-Solution)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
12	x	No	None	0.392	-1.2	0.05	2	cold	2 ml	No	mechanical shaking, 1 min	$SnCl_2$/HCl, 1 h, 80 °C	H_2O/HCl	HCl		No	No	GC-MSD	StAdd-SP	No	StAdd-SP				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
15		Yes	> 2 y	0.435	-0.9	0.0005		cold	No		mechanical shaking	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-Ion Trap, standard addition	StAdd-SP	No	StAdd-SP	108 % (0.2 mg/kg)	SB-EUPT	4	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type), weight 0.5 g of sample
16		Yes	> 2 y	0.832	2.0	0.01	50	cold			manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.; Iso-octane	GC-MSD, m/z: 76 and 78	PS-ML	No	None	82 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
19		Yes	> 2 y	0.65	0.6	0.05	25	ambient	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	NAOH+ H_2SO_4	Spectrophotometer, 272+302+332nm	PS-ML	No	None	100 % (0.050 + 0.50 mg/kg Thiram)	SB-other	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
20		Yes	> 2 y	0.5	-0.4	0.05	1	ambient	8 ml	No	mechanical shaking, 15 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No		GC-MSD		No	StAdd-SP	70 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
21		Yes	> 2 y	0.883	2.3	0.01	25	cold	Yes	No	mechanical shaking	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MSD	PS-ML	No		121 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavge step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
22		Yes	> 2 y	0.704	1.0	0.05	10	ambient	hydrolysis sln	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, 2 h, 70 °C	H_2O/HCl	1× (HCl, pH 1)		No		GC-(μ) ECD	MM-ML	No	None	99 % (CS_2, 0.5 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
23	x	Yes	1 - 2 y	0.21	-2.5	0.05	200		No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No	None	76 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
25		Yes	> 2 y	0.648	0.6	0.05	10	deep frozen	10 ml	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	83 % (0.411 mg/kg Thiram)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, headspace sampling of non-polar solvent
26	x	No	> 2 y	0.325	-1.7		50	ambient	45 mL	after H_2O, 15 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-(P) FPD	PS-ML	No		86.7 %		3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
27		Yes	> 2 y	0.27	-2.1	0.02	2	ambient	Yes	No	mechanical shaking, 15 min	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MSD	StAdd-SP	other compound	StAdd-SP		SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
28		Yes	> 2 y	0.498	-0.4	0.05	50		50 ml	after H_2O, 5 min	45 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 50 ml	HCl		Yes		Spectrophotometer	MM-ML	No	None	76 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
29	x	Yes	> 2 y	0.558	0.0	0.05	25	slightly frozen	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 25 ml	HCl		No	Dessication with Na_2SO_4	GC-(μ) ECD, Agilent	MM-ML	No	None	93 % (0.6 mg/kg)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
30		Yes	> 2 y	0.228	-2.4	0.01	100	ambient	200 mL		60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer, 272 nm	PS-ML	No	None	98 % (0.15 mg/kg spiking lvel)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
31		Yes	> 2 y	0.46	-0.7	0.01	3	ambient	3 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	HS-CG-MS	PS-ML	No		62.5 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
32		No	> 2 y	0.86	2.2	0.2	50	just thawed	200 mL	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None			1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
33		Yes	> 2 y	0.484	-0.5	0.05	10	ambient	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	iodoethane					$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
34		Yes	> 2 y	0.688	0.9	0.5	50	deep frozen			manual shaking, 1 min	$SnCl_2$/HCl	H_2O/ $SnCl_2$/HCl, 200 mL	1× (HCl)		Yes	No	Spectrophotometer, UV 435 nm	MM-ML	No	None		SB-EUPT	1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
35		Yes	> 2 y	0.568	0.1	0.05	50	ambient	45 mL	No	manual shaking, 60 min	$SnCl_2$/HCl, water bath, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	PS-ML	No	None	83 % (0.550 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
37	x	Yes	> 2 y	0.28	-2.0	0.01	13	ambient	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD, SIM	PS-ML		None	96 % (Hydrolysis recovery with Thiram)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
38		Yes	> 2 y	0.696	1.0	0.05	25	ambient	25 ml	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(μ) ECD	MM-ML	No	StAdd-SP	87.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
40		Yes	> 2 y	0.541	-0.1	0.05	20	ambient	Yes	after H_2O, 10 min	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Destillation	Spectrophotometer	PS-ML	No	None	92.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type); result not blank corrected
41	x	No	None	0.334	-1.6	0.05	4	ambient	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-(S) FPD	PS-ML	No	StAdd-SP	75 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
43		Yes	> 2 y	0.87	2.2	0.02	1	ambient	Yes	No	mechanical shaking, 60 min	$SnCl_2$/HCl, 80 °C	H_2O/HCl	HCl		No	No	GC-Ion Trap	MM-ML	No	PrCal				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Dithiocarbamates

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavge step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
44	x	Yes	> 2 y	0.393	-1.2	0.05	25	ambient	40 ml	after H_2O, 5 min	ultrasonic bath, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD		No	StAdd-SP				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
45		Yes	> 2 y	0.661	0.7	0.02	20	cold	No	No	60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 100 ml	HCl		No	No	GC-(P) FPD	StAdd-SP	No	None	104 % (0.3 mg/kg spiking level)	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
46		Yes	> 2 y	0.496	-0.5	0.01	25	ambient	No	No	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	No	None	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
47	x	Yes	> 2 y	0.544	-0.1	0.05	50	ambient	No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	81.2 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
48		Yes	> 2 y	0.881	2.3	0.1	25	ambient	see hydrolysis	No	manual shaking, 5 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)		No	Centrifugation	GC-MSD	PS-ML	No	None	93 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, GC-MS
49	x	No	> 2 y	0.53	-0.2	0.05	25	cold	No		mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane, toluene	HCl		No	Liq-liq part.	GC-MSD	PS-ML	No	None	62 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, no H_2O adittion
50		Yes	> 2 y	0.465	-0.7	0.05	25	slightly frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No		GC-(P) FPD	PS-ML	No	None	76 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
51	x	Yes	> 2 y	0.706	1.1	0.05	30	slightly frozen	No		mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No		GC-MSD	PS-ML	No		86 %	SB-EUPT	4	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
53		No	> 2 y	0.92	2.6	0.05	2	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC/ECD	MM-ML	No	PrCal				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
54	x	Yes	> 2 y	0.432	-0.9	0.125	10	ambient	15 ml	after H_2O, 5 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD, Agilent 7000c QQQ	PS-ML	No	None	57 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, isooctane
55		Yes	> 2 y	0.568	0.1	0.02	50	deep frozen	45 mL H_2O	after H_2O, 20 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Centrifugation	GC-MS/MS (QQQ)	PS-ML	No	None	78 % (0.04 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
56		Yes	> 2 y	0.451	-0.8	0.04	50					$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	102 % (Spiking at LOQ)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
57		No	> 2 y	0.452	-0.8	0.01	5	ambient	Yes	No		$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MSD	MM-ML	chloroform	None	101 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
58		Yes	> 2 y	0.328	-1.7	0.05	2	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No		GC-MSD	StAdd-SP	thiophene	StAdd-SP			3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
59	x	Yes	> 2 y	1.064	3.6	0.05	25	cold	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(μ) ECD	PS-ML	No	None	109 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
61		Yes	> 2 y	0.7	1.0	0.01	3	cold	No		> 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-(μ) ECD	MM-ML	No	PrCal	117 %	QC		$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
63		Yes	> 2 y	1.03	3.4	0.1	2	ambient	No	No	manual shaking, 10 min	HCl/$SnCl_2$,	H_2O/HCl	HCl			No	GC-FID		other compound	PrCal	85 %	QC		$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
64	x	Yes	> 2 y	1.03	3.4	0.05	25	deep frozen	No	No		$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MSD	MM-ML	No	None	110 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
67	x	Yes	> 2 y	0.894	2.4	0.025	5	ambient	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, water bath, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl		No		GC-Ion Trap	PS-ML	No		100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
68		Yes	> 2 y	0.63	0.5	0.05	200		200 mL			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No		84.4 %	SB-EUPT	2	PN-EN 12396-1: SnCl2/HCL-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavge step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
70	x	Yes	> 2 y	0.589	0.2	0.2	25	ambient	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-(P) FPD	MM-ML	No	None	80 %	SB-other	5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
72		Yes	> 2 y	0.653	0.7	0.05	50	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Pb(CH3COO)2	Spectrophotometer	PS-ML	No	None	81 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
73		Yes	> 2 y	0.599	0.3	0.05	10	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-(P) FPD	PS-ML	thiophene	None	95.6 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
74	x	Yes	> 2 y	0.91	2.5	0.05	50	cold	Yes	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	$^{13}CS_2$	None				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
75		Yes	> 2 y	0.459	-0.7	0.05	25	ambient	see Solvent 1	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Distillation passing through NaOH+ H_2SO_4	Spectrophotometer	PS-ML	No	None	87 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
76		Yes	1 - 2 y	0.89	2.4	0.05	25	ambient	Yes	No		$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)				GC-(μ) ECD	PS-ML	chloroform	None	76 %			$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, Analysis of Dithiocarbamate Residues in Foods of Plant Origin Involving Cleavage into Carbon Disulfide, Partitioning into Isooctane
78		Yes	> 2 y	0.81	1.8	0.01	2	ambient	No	No	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MS/MS (QQQ)	PS-ML	generic IS	None	94 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
81	x	Yes	> 2 y	0.616	0.4	0.05	50	cold	No		ultrasonic bath, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl 25 ml isooctane	HCl		No		GC-MSD	MM-ML	No	None	80 %	SB-other	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
82		Yes	> 2 y	0.526	-0.2	0.1	50	ambient	No		30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	gaswashing	Spectrophotometer	PS-ML	No	None			4	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
84	x	Yes	> 2 y	0.762	1.5	0.05	25	slightly frozen	No	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	No		No	No	GC- (P) FPD, GC-(S) FPD	PS-ML	No	No	85 %	SB-EUPT	1	SnCl2/HCL-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS2
85		No	> 2 y	1.25	4.9	0.05	5	deep frozen	No	No	manual shaking, 5 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC- (P) FPD	MM-ML	No	(Leer)	97 % (0,05 mg/kg)	QC	2	SnCl2/HCL-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS2
86		Yes	> 2 y	0.617	0.4	0.05	20	slightly frozen	No	No	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	78 %	QC	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
87		Yes	> 2 y	0.563	0.0	0.02	50	just thawed	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer, 272 nm, 302 nm, 332 nm	PS-ML	No	None	90.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
88		Yes	> 2 y	0.547	-0.1	0.02	50		No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No		87 % (thiram)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
89		Yes	< 1 y	0.52	-0.3	0.05	10	ambient	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	MM-SL	No	PrCal	75 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
90		Yes	1 - 2 y	0.423	-1.0	0.05	10	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	Dichlormethane	None	63 % (0.5 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
91	x	Yes	> 2 y	0.665	0.8	0.05	10	slightly frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-(P) FPD (following derivatization)	PS-ML	thiophene	None	68.8 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
93		Yes	> 2 y	0.45	-0.8	0.05	100		200 mL	after H_2O, 10 min		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	83.4 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavge step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
94	x	Yes	> 2 y	0.292	-1.9	0.01	25	deep frozen	25 g	No		$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	No	None	40.8 % (0.5 mg/kg (with maneb))	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2, GC-MS used (m/z 76)
95		No			-3.6 (FN)	0.05	50	ambient	No		> 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MS/MS (QQQ)	PS-ML						$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
98	x	Yes	> 2 y	0.509	-0.4	0.01	10	deep frozen	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-MSD	MM-ML	No		75 %	SB-EUPT	> 5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
99		Yes	> 2 y	0.59	0.2	0.3	75	ambient	No			$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	70 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
100		Yes	> 2 y	0.05622	-3.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	$SnCl_2$/HCl	ACN	HCl		No	No	GC-MS/MS (QQQ)	MM-ML	CS_2-^{13}C	None				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
102		Yes	> 2 y	0.55	-0.1	0.5	10		No	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	StAdd-SP	90 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
103		Yes	> 2 y	0.55	-0.1	0.3	100	ambient	Yes	after H_2O, 10 min		$SnCl_2$/HCl	H_2O/HCl	HCl				Spectrophotometer, UV	PS-ML	No	None	92 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
104		No	< 1 y	0.508	-0.4	0.04	50	deep frozen	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	96 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
106		Yes	> 2 y	0.63	0.5	0.05	25	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	105 % (matrix EUPT SRM7)	SB-other	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
107		Yes	> 2 y	0.51	-0.4	0.05	2	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part., Filtration	GC-(P) FPD, Sulfur Filter	MM-ML	No	None	85 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
108		Yes	> 2 y	0.47	-0.6							$SnCl_2$/HCl	H_2O/HCl					no data		No		83 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
109		Yes	> 2 y	0.196	-2.6	0.01	5	ambient	10 ml	No	ultrasonic bath, 60 min	$SnCl_2$/HCl	$SnCl_2$/HCl	HCl				GC-(μ) ECD	MM-ML	No					$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
112		No	None	0.47	-0.6	0.1	50	slightly frozen	45 mL	after H_2O, 5 min	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-TOF	MM-ML	No	RecF	85 %	SB-EUPT	4	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
113		Yes	> 2 y	0.65	0.6	0.05	25	cold				$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	MM-ML	No	None	85 % (0.05. Thirum)	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
114		No		0.84	2.0	0.05														other compound	None	83 %	SB-EUPT	1	no data
115		Yes	None	0.66	0.7	0.025	5	deep frozen	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Centrifugation, Liq-liq part	GC-MS	PS-ML	generic IS	None	100 %		> 5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
116		Yes	> 2 y	0.88	2.3	0.5	6	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	PS-ML	No	None	80 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
117		Yes	> 2 y	0.392	-1.2	0.01	25	just thawed	Yes	after H_2O, min	manual shaking	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	68 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
118		Yes	> 2 y	0.721	1.2	0.01	1	ambient	No	No		$SnCl_2$/HCl	$SnCl_2$/HCl	HCl				GC-MSD, HeadSpace sampling with GC/MS		No	None			4	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Ethephon

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.151	-0.3	0.02	10	deep frozen	No	No	mechanical shaking, 60 min	No	MeOH / H_2O / FA	No		No	Disp.-SPE; carbon	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	85 % (0.2 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.148	-0.3	0.05	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.215	1.3	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.13	-0.8	0.02	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	Yes	> 2 y	0.157	-0.1	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
8		No	> 2 y	0.025	-3.4	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		FA				No	LC-MS/MS (QQQ)		ILIS	other	71 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
10		Yes	> 2 y	0.185	0.6	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	None	104 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.162	0.0	0.02	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	MM-ML	ethephon-D_4		99 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
12	x	Yes	> 2 y	0.169	0.2	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	StAdd-EA	105.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.162	0.0	0.02	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	87 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.193	0.8	0.02		ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	90 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), weight 2.5 g of sample
16		Yes	> 2 y	0.139	-0.6	0.05	5	cold	9.3 mL	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	108.4 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.178	0.4	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		ethephon-D_4	StAdd-SP	88 %	SB-EUPT	2	Accreditation in request
21		Yes	> 2 y	0.159	-0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
22		No	None	0.193	0.8	0.05	10	ambient	140 ml	No	mechanical shaking, > 60 min	No		1× (pH 14)		Yes		GC-FID (following derivatizsation)	MM-ML	Aceton	None	102 % (0.2 mg/kg)	SB-EUPT	1	Method involv. ethylene-release (§ 64 LFGB 00.00-47-type); GC-Headspace
23	x	Yes	1 - 2 y	0.158	-0.1	0.02	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	93 %	SB-EUPT	3	other (EN 15055, 2006-08)
25		Yes	> 2 y	0.139	-0.6	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	PrCal	117 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
26	x	No	1 - 2 y	0.388	5.6		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	104.2 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	> 2 y	0.153	-0.2	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	100 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
28		No	1 - 2 y	0.164	0.1	0.02	5		10 ml	after H_2O, 5 min	mechanical shaking, 5 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	73 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
29	x	Yes	< 1 y	0.218	1.4	0.05	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), Agilent	MM-ML	ethephon-D_4	PrCal	102 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
30		Yes	> 2 y	0.141	-0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ), 143/107	PS-ML	ethephon-D_4	PrCal	80 % (96 % D_4-Ethephon recovery)	SB-EUPT	1	extraction with MeOH, LC-MS/MS detection
31		Yes	< 1 y	0.047	-2.8	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	perclorarte ^{18}O	PrCal	100 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.21	1.2	0.02	10	ambient	No	No	mechanical shaking, 10 min	No	ACN, MeOH 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4					in house
34		Yes	> 2 y	0.137	-0.6	0.05	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	PrCal	111 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
35		Yes	> 2 y	0.24	1.9	0.02	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	133 % (0.23 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
36		Yes	> 2 y	0.411	6.2	0.02	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_4 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.224	1.5																				
40		Yes	> 2 y	0.128	-0.8	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		No	None	0.15	-0.3	0.05	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
44	x	Yes	> 2 y	0.351	4.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		Yes	> 2 y	0.148	-0.3	0.02	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	107 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.168	0.2	0.01	5	ambient	10 ml	No	mechanical shaking, 60 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	113 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
47	x	No	None	0.155	-0.2	0.1	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Freezing out	LC-MS/MS (QQQ)	MM-SL	No	None	72.1 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
51	x	Yes	> 2 y	0.185	0.6	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		106 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
55		Yes	< 1 y	0.27	2.7	0.02	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	PrCal	107 % (0.04 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
57		No	> 2 y	0.221	1.5																				
58		No	1 - 2 y	0.146	-0.4	0.02	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
60	x	No	> 2 y	0.0677	-2.3		5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	87 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.15	-0.3	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	101 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	0.15	-0.3	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	80 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
64	x	No	> 2 y	0.196	0.8	0.04	5	deep frozen	10 ml	after H_2O, 10 min		No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	299.8 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.215	1.3	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	71 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
67	x	No	None	0.1238	-0.9	0.05	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		102 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.152	-0.2	0.05	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	100 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
72		No	1 - 2 y	0.146	-0.4	0.02	10	ambient	No	after H_2O, 5 min	mechanical shaking, 5 min	No	Isooctane	No		derivated by diazomethane	No	GC-(P) FPD (following derivatization)	MM-ML	No	None	77 %	SB-EUPT	3	other (derivated by diazomethane)
74	x	Yes	> 2 y	0.241	2.0	0.05	5	cold	Yes	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
75		No	None	0.082	-2.0	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4	StAdd-SP	115 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
77		No	> 2 y	0.059	-2.5	0.01												no data		No	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
78		Yes	> 2 y	0.083	-1.9	0.01	5	ambient	No	No	ultra turrax, 1 min	No	EtOAc	1×		Yes	Dessication with $MgSO_4$	GC-MS/MS (QQQ) (following cleavage)	MM-ML	generic IS	None	115 %	SB-EUPT	1	Method involv. ethylene-release (§ 64 LFGB 00.00-47-type)
81	x	Yes	> 2 y	0.137	-0.6	0.02	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	None	95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.2	0.9	0.05	5	ambient	10 ml	No	manual shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
84	x	Yes	> 2 y	0.212	1.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (as for QuPPe)		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	106 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
85		No	1 - 2 y	0.156	-0.1	0.02	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		119 % (0.02 mg/kg)	QC	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		No	< 1 y	0.133	-0.7	0.06	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	85.9 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:20
89		Yes	< 1 y	0.194	0.8	0.01	5	ambient	25 ml	after H_2O, 5 min	mechanical shaking, 10 min		DCM	1× (FA)				LC-MS/MS (QQQ)	MM-ML	ioxynil	PrCal	60 %	SB-EUPT	3	other (H_2O / DCM)
91	x	No	> 2 y	0.187	0.6	0.02	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), 1 daughter ion	MM-ML	No	None	78.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no ISTD was used
94	x	No	None	0.133	-0.7	0.1	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	78.2 % (0.1 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.07	-2.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.23061	1.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	56.99 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
103		Yes	1 - 2 y	0.124	-0.9	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 10 min		MeOH + 1 % FA	No			No	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	72 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
113		Yes	> 2 y	0.083	-1.9	0.05	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	76 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
115		Yes			-3.5 (FN)	0.5	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML						QuEChERS-Citrate buffered (EN 15662), modified method
116		Yes	1 - 2 y	0.28	2.9	0.02	2	ambient	No	No	mechanical shaking, 5 min	No	ACN, H_2O	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	102 %	SB-EUPT	1	other (extraction with ACN:H_2O)
117		Yes	> 2 y	0.114	-1.2	0.01	2	just thawed	Yes	after H_2O, min	manual shaking		Isooctane					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77 %	SB-EUPT	1	O-tins: QuEChERS-based mth by EURL-SRM
118		No	None	0.0032	-3.9		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, negative	MM-SL	glyphosate-^{13}C	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	1 - 2 y	0.697	0.9	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	1× (Borate buffer)		FMOC-Cl	Liq-liq part.; EtOAc/CyH 1/1	LC-MS/MS (QQQ)	MM-ML	ILIS	StAdd-SP	98 %	SB-EUPT	1	Method involv. deriv. w. FMOC
2		Yes	1 - 2 y	0.616	0.3	0.05	3	deep frozen	9.75 mL	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	PS-ML	glyphosate $^{13}C^{15}N$	None	101 % (0.5 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Cf. extraction, clean up and chromatography
3		Yes	1 - 2 y	0.506	-0.4	0.04	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	108 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.527	-0.3	0.05	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.55	-0.1	0.05	1	ambient	10 ml	No	mechanical shaking, 60 min	Yes	H_2O	No		FMOC-Cl	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	Method involv. deriv. w. FMOC
6	x	Yes	> 2 y	0.568	0.0	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
8		No	> 2 y	0.262	-2.2	0.05	1	ambient	9 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	84 %	SB-EUPT	2	Method involv. deriv. w. FMOC
10		Yes	> 2 y	0.639	0.5	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	108 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.421	-1.0	0.05	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$		120 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
12	x	Yes	> 2 y	0.582	0.1	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-EA	103.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
13		Yes			-3.6 (FN)	0.01												no data							Method involv. deriv. w. FMOC
14		No	None	0.775	1.5																				
15		Yes	> 2 y	0.64	0.5	0.04		ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	96 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), weight 2.5 g of sample
16		Yes	1 - 2 y	0.576	0.1	0.05	3	ambient	25 + 25 mL	No	ultrasonic bath, 10 min	No	H_2O, DCM: 2-propanol	2× (alkaline/ acidic)		FMOC	DCM:2-propanol	LC-MS/MS (QQQ)	MM-ML	glyphosate-^{13}C	PrCal	107.2 %	SB-EUPT	2	Method involv. deriv. w. FMOC
18		Yes	> 2 y	0.62	0.4	0.05	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		90 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.55	-0.1	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		glyphosate 2	StAdd-SP	110 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
21		Yes	> 2 y	0.453	-0.8	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	93 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
22		Yes	> 2 y	0.607	0.3	0.05	10	ambient	No	No	ultrasonic bath, 20 min	No	DCM, part. with 20 ml HCl, 10 ml DCM	1× (neu-tralisation with NaOH to pH 6-8)		FMOC	Centrifugation; ion-exchange	LC-FLD (Fluorescence)	MM-SL	No	PrCal	rec. spiking level = calibr. level, (deri-vatis. step included, rec. figure not posible)	SB-EUPT	3	Method involv. deriv. w. FMOC, acid extraction,neutralisation,ion-exchange,LC-Fluorescense-Detection after derivatisation with FMOC

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
23	x	Yes	1 - 2 y	0.788	1.6	0.05	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	96 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	> 2 y	0.623	0.4	0.02	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	PrCal	103 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
26	x	No			-3.6 (FN)	0.5	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		FMOC	No	LC-MS/MS (QQQ)	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	> 2 y	0.67	0.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	109 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
28		Yes	> 2 y	0.651	0.6	0.05	5		10 ml	after H_2O, 5 min	mechanical shaking, 5 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	72 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
29	x	Yes	1 - 2 y	0.619	0.4	0.01	5	slightly frozen	No		manual shaking, 2 min		H_2O, DCM, H_2O+0.1 %FA	1× (pH 6)		FMOC	SPE-column; Oasis HLB	LC-MS/MS (QQQ), Agilent	MM-ML	glyphosate $^{13}C^{15}N$	PrCal	101 %	SB-EUPT	4	Method involv. deriv. w. FMOC
30		Yes	> 2 y	0.564	0.0	0.01		ambient	No		mechanical shaking, 30 min	No	0.1 mol/l HCl	1×		after cleanup derivatisation with FMOC	Centrifugation; Cleanup 2 with Dichlormethan	LC-MS/MS (QQQ), 390/168	PS-SL	$^{13}C,^{15}N$ glypho-sate	PrCal	88 % (88 % ^{13}C. ^{15}N Glyphosat recovery)	SB-EUPT	1	Method involv. deriv. w. FMOC, C18 clean up
32		No	> 2 y	0.34	-1.6	0.05	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.62	0.4	0.05	3	ambient	No	No	mechanical shaking, 60 min	No	H_2O 0.1 FA, H_2O 1 % FA	No		adding FMOC and Borate buffer	SPE-column (C18)	LC-MS/MS (QQQ)	StAdd-SP	No					Method involv. deriv. w. FMOC
34		Yes	> 2 y	2.85	16.1	0.04	5	deep frozen	No		mechanical shaking, 15 min	No	H_2O, 20 mL	No		FMOC	Liq-liq part.; EtOAc	LC-MS/MS (QQQ), ESI neg	MM-ML	No	PrCal	73 %	SB-EUPT	1	Method involv. deriv. w. FMOC
35		Yes	> 2 y	0.598	0.2	0.02	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	glyphosate-^{15}N-^{13}C	None	110 % (0.559 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
36		Yes	> 2 y	0.676	0.8	0.05	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.495	-0.5	0.05	3	ambient	25 ml	No	ultrasonic bath, 10 min	No	H_2O	No		No	No	LC-MS	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	lab method
40		Yes	> 2 y	0.593	0.2	0.1	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		Yes	1 - 2 y	0.229	-2.4	0.2	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
44	x	Yes	> 2 y	0.592	0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		No	1 - 2 y	0.241	-2.3	0.05	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	93 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.592	0.2	0.05	5	ambient	10 ml	No	mechanical shaking, 60 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	106 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
48		Yes	> 2 y	0.406	-1.1	0.03	20	ambient	see pH adjust-ment	No	mechanical shaking, 30 min	No	DCM	more than 2× (HCl/HCl/ buffer)		OPA	Centrifugation, SPE-column (ion exchange)	LC-FLD (Fluorescence)	PS-ML	No	None	86 %	SB-EUPT	2	Method involv. post-colum deriv. w. OPA (DFG-405 type)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
51	x	Yes	> 2 y	0.526	-0.3	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	ILIS	StAdd-EA	87 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
52		Yes	1 - 2 y	0.602	0.2	0.05	5	just thawed	10 ml	after H_2O, 10 min	mechanical shaking, 30 min	No		No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N		101.2 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), 0.5 % FA in Eluent A and B, freezing out of MeOH extract
54	x	Yes	> 2 y	0.469	-0.7	0.02	1	ambient	No	No	ultra turrax, 1 min	No	H_2O, Isooctane, DCM	No		FMOC	Centrifugation, Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	other	92 %	SB-EUPT	1	Method involv. deriv. w. FMOC, Goscinny et al, Food Analytical Methods 2012;5;1177-1185
55		Yes	> 2 y	0.53	-0.3	0.01	3	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	Isooctane, DCM	1× (pH 9)		FMOC	SPE-column (C18)	LC-MS/MS (QQQ)	MM-ML	glyphosate $^{13}C^{15}N$	PrCal	103 % (0.02 mg/kg)	SB-EUPT	1	Method involv. deriv. w. FMOC
57		No	1 - 2 y	0.584	0.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	66 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	1 - 2 y	0.653	0.6	0.05	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
60	x	No	> 2 y	0.484	-0.6	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.3	-1.9	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	92 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	0.391	-1.2	0.1	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	55 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial
64	x	No	> 2 y	0.746	1.3	0.08	5	deep frozen	10 ml	after H_2O, 10 min		No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	107 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.612	0.3	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	88 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.772	1.4	0.5	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	ILIS	None	97 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
72		No	1 - 2 y	0.264	-2.1	0.05	25	ambient	No	No	mechanical shaking, 20 min	No	H_2O	No		derivated by TMOAc	No	GC-(P) FPD (following derivatization)	PS-ML	No	RecF	53 %	SB-EUPT	3	other (derivated by TMOAc)
74	x	Yes	> 2 y	0.55	-0.1	0.01	3	cold	Yes	No	ultrasonic bath, 20 min	No	H_2O	No		FMOC	Liq-liq part.	LC-MS/MS (QQQ)	MM-ML	1,2-$^{13}C_2$ ^{15}N-glyphosate	None				Method involv. deriv. w. FMOC
75		Yes	> 2 y	0.517	-0.4	0.05	2	ambient	see solvent details	after H_2O and organic solvent, 30 min	ultrasonic bath, 30 min	No	EtOAc, H_2O/MeOH/Borat-buffer 6:3:1	1× (after SPE-elution to pH 9 with NH_3)		with fluorenyl-methyl)-chlorformiat	SPE-column; Oasis MAX, 30 Âµm; elution with ACN/HCl	LC-MS/MS (QQQ)	StAdd-SP	glyphosate 1,2-$^{13}C_2$ ^{15}N	StAdd-SP	106 %	SB-EUPT	2	Method involv. deriv. w. FMOC
78		Yes	> 2 y	0.42	-1.0	0.05	3	ambient		No	ultra turrax, 1 min	No	Isooctane, H_2O	No		Yes	Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS	None	90 %	SB-EUPT	1	Method involv. deriv. w. FMOC
81	x	Yes	> 2 y	0.603	0.2	0.05	5	cold	9.5 ml		mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	glyphosate (^{13}C, ^{15}N)	None	103 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.462	-0.7	0.1	5	ambient	10 ml	No	manual shaking, 5 min	No	Isooctane	2× (ammonia, FA)		deriv. w. isobutyl-chloroformate	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	Method involv. deriv. w. FMOC
83		Yes	> 2 y	0.707	1.0	0.04	25	ambient			mechanical shaking, 30 min	No	H_2O/HCl, DCM, 0.1M HCl	1× (pH between 1.6 and 2.4)			SPE-column (ion exchange), SPE-column (ion exchange), SPE 1 = Chelex 100; SPE2 = AG 1-X8	LC-FLD (Fluorescence), postcolumn derivatisation (OPA)	PS-ML	No	RecF	50 %	SB-EUPT	3	Method involv. post-colum deriv. w. OPA (DFG-405 type)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Glyphosate

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
84	x	Yes	> 2 y	0.652	0.6	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (as for QuPPe)		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
89		No	< 1 y	0.442	-0.9	0.05	5	ambient	25 ml	after H_2O, 5 min	mechanical shaking, 10 min		DCM	1× (FA)				LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	88 %	SB-EUPT	3	other (H_2O / DCM)
91	x	No	> 2 y	0.242	-2.3	0.05	20	slightly frozen	No		ultra turrax, 5 min	No	H_2O, 100 ml H_2O	No		with triflouoro-aceticacid anhydride and fluorated buthanole	Liq-liq part., SPE-column (ion exchange), LLE with DCM	LC-MS/MS (QQQ), 4 daughter ions	PS-ML	No	None	66.5 %	SB-EUPT	1	other (with derivatization), 1) extraction with H_2O, 2) clean-up, 3) derivatization
94	x	No	None	0.472	-0.7	0.1	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	44.6 % (0.1 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.536	-0.2		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.84408	1.9	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	72.8 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
106		No	< 1 y	0.67	0.7	0.5	1	deep frozen										LC-Orbitrap	MM-ML	ILIS	None	99 % (matrix EUPT SRM7)	SB-other	1	Method involv. deriv. w. FMOC
111		Yes	> 2 y	0.597	0.2	0.05	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	96 % (2x MRRL)	SB-EUPT	1	other
113		Yes	> 2 y	2.494	13.6	0.05	2	ambient										LC-MS/MS (QQQ)	StAdd-SP	No	StAdd-SP	100 %			Method involv. deriv. w. FMOC; detected in blank at high concentration.
114		Yes		0.58	0.1	0.05														ILIS	StAdd-SP	120 %	SB-EUPT	1	no data
115		Yes			-3.6 (FN)	0.01	1	deep frozen	10 ml	No	mechanical shaking, 20 min	No	DCM, H_2O	2× (sodium borate)		FMOC over-night	Centrifugation, SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML						Method involv. deriv. w. FMOC
116		Yes	> 2 y	0.52	-0.3	0.1	2	ambient	No	No	ultra turrax, 1 min	No	H_2O	No		FMOC	No	LC-MS/MS (QQQ)	PS-SL	No	None	73 %	SB-EUPT	2	Method involv. deriv. w. FMOC
118		No			-3.6 (FN)		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, negative	MM-SL						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No			-3.5 (FN)	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML				SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.061	-1.0	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML	$MCPP-D_3$	PrCal	98 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.09	0.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	116 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.125	2.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	$MCPA D_3$	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	2.5-dichloroben-zoic acid	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.081	0.0	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)		ILIS	other	83 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
10		Yes	> 2 y	0.0957	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	$MCPA D_6$	None	95 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		Yes	> 2 y	0.082	0.0	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		105 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
12	x	Yes	1 - 2 y	0.091	0.5	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	105.7 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
15		Yes	> 2 y	0.0804	0.0	0.004	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	93 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
16		Yes	> 2 y	0.0612	-1.0	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	63.5 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
18		Yes	> 2 y	0.058	-1.1	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.09	0.4	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0815	0.0	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	76 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		Yes	> 2 y	0.082	0.0	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	98 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
22		Yes	> 2 y	0.0891	0.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4), PSA/$MgSO_4$ (pH > 8))		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	98 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
23	x	Yes	1 - 2 y	0.1	0.9	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	110 %	SB-EUPT	3	other

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
24	x	No	> 2 y	0.0294	-2.6	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
25		Yes	> 2 y	0.087	0.3	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	85 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
26	x	No	> 2 y	0.08	-0.1		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	70.8 %		3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.115	1.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	106 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
28		No	> 2 y	0.068	-0.6	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	106 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.081	0.0	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	95 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
30		Yes	> 2 y	0.075	-0.3	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabu-tyl-ammoni-umhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:214/216/141	PS-SL	No	None	88 %	SB-EUPT	1	alkaline hydrolysis extraction, GPC, acid/base distribution, methylation, GC-MSD detection
31		Yes	< 1 y	0.075	-0.3	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	46 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
32		Yes	> 2 y	0.068	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.085	0.2	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
34		No	> 2 y	0.016	-3.2	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Centrifugation	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	76 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
35		Yes	> 2 y	0.0688	-0.6	0.01	5	ambient	10 ml	No	mechanical shaking, 30 min	No	EtOAc with 1 % HOAc	1× (1 % HOAc)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	80 % (0.101 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time
36		Yes	> 2 y	0.0964	0.8	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	RecF	68 % (Avegrage from ongoing AQC samples)	QC	> 5	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
37	x	Yes	> 2 y	0.079	-0.1	0.05	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	102 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA cleaning
38		Yes	> 2 y	0.081	0.0	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	87.8 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
40		Yes	> 2 y	0.074	-0.4	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifu-gation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	80 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.08	-0.1	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA				QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	> 2 y	0.102	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
45		Yes	> 2 y	0.0795	-0.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	99 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
46		Yes	1 - 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	93 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
47	x	No	None	0.084	0.1	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	80.6 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.091	0.5	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	101 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.081	0.0	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	104 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.073	-0.4	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, Ammonium Acetate	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	90 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 with extract conc
55		Yes	> 2 y	0.11	1.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-dimethyl-phenoxy) HOAc	PrCal	88 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
56		Yes	> 2 y	0.0741	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	91 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.072	-0.4	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	129 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
58		Yes	> 2 y	0.061	-1.0	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
59	x	Yes	> 2 y	0.071	-0.5	0.05	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min	NaOH	ACN	1× (H_2SO_4)		Yes	C18/$MgSO_4$	GC-MSD (following derivatization)	MM-ML	No	None	75 %	SB-EUPT	2	other (with derivatization)
60	x	No	< 1 y	0.0961	0.7	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	95 %	SB-EUPT	3	Modified QuEChERS
61		Yes	> 2 y	0.1	0.9	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	97 %	QC		QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.122	2.0	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	45 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
64	x	Yes	> 2 y	0.038	-2.1		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	40.4 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
65	x	Yes	> 2 y	0.0656	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	69 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	1 - 2 y	0.091	0.5	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	98 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.1076	1.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		89 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
70	x	No	> 2 y	0.0767	-0.2	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	60 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
72		Yes	> 2 y	0.0798	-0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
73		No	None	0.0912	0.5	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	94.9 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.066	-0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
75		Yes	> 2 y	0.064	-0.8	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	MCPA D_6	StAdd-EA	63 %	QC	> 5	A-QuEChERS (with 1 % FA)
76		Yes	1 - 2 y	0.073	-0.4	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No				LC-Orbitrap	PS-ML	2.4,6-trichloro-phenol	None	82 %			A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.135	2.7	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
78		Yes	> 2 y	0.047	-1.7	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifuga-tion	LC-MS/MS (QQQ)	PS-ML	generic IS	None	65 %	SB-EUPT	2	in house method
79		No	< 1 y	0.096	0.7	0.05	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	107 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
80		Yes	> 2 y	0.083	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.0741	-0.3	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	80 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
82		Yes	> 2 y	0.078	-0.2	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	None			4	QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.078	-0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
84	x	No	None	0.079	-0.1	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
85		No	> 2 y	0.07	-0.5	0.01	5	cold	10 ml	after H_2O and organic solvent, 30 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		80 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
87		Yes	> 2 y	0.086	0.2	0.01	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	70.6 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
89		Yes	< 1 y	0.057	-1.2	0.005	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	84 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
90		Yes	> 2 y	0.0805	0.0	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	92 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
91	x	Yes	> 2 y	0.0752	-0.3	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.7 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
94	x	Yes	> 2 y	0.0963	0.7	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	103 % (0.01 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
97	x	No	> 2 y	0.053	-1.4	0.02	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	nicarbazin	None	74 % (0.02 mg/kg)	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM, EURL SRM

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
98	x	Yes	1 - 2 y	0.087	0.3	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		98 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.11445	1.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	133.61 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.087	0.3	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	68 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
106		Yes	> 2 y	0.079	-0.1	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	93 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
107		Yes	> 2 y	0.0792	-0.1	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	0.104	1.1	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	110 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
114		No		0.029	-2.6	0.02														other compound	StAdd-SP	104 %	SB-EUPT	1	no data
115		Yes	None	0.02	-3.0	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
116		No	1 - 2 y	0.09	0.4	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
118		No	None	0.0667	-0.7		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Mepiquat

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	> 2 y	0.112	-0.1	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	21 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
2		Yes	> 2 y	0.122	0.3	0.01	10	deep frozen	No	No	mechanical shaking, 60 min	No	H_2O / MeOH / HCl	No		No	Disp.-SPE; alumina	LC-MS/MS (QQQ)	PS-ML	mepiquat D_3	None	89 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.101	-0.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.124	0.4	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	mepiquat D_3	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.11	-0.1	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	Yes	> 2 y	0.115	0.0	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		No	> 2 y	0.093	-0.7	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	85 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
9		Yes	> 2 y	0.114	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				Filtration	LC-MS/MS (QQQ)	PS-ML	isotope D_3	PrCal	106 % (not corrected)	SB-EUPT	3	SIST EN 15055:2006
10		Yes	> 2 y	0.139	0.9	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.1	-0.5	0.01	10	deep frozen	10 ml	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ)	MM-ML	D_3-mepiqua-tiodid		102 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	other
12	x	Yes	> 2 y	0.106	-0.3	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	StAdd-EA	94.8 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.101	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.116	0.1	0.004	5	ambient	No	after H_2O and organic solvent, 10 min	mechanical shaking, 20 min	No		1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	79 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), extraction mixture: 20 mL 80 % ACN in H_2O, 1 % FA
16		Yes	< 1 y	0.115	0.0	0.01	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	mepiquat iodide D_3	None	114.2 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
18		Yes	> 2 y	0.09	-0.8	0.01	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
19		Yes	1 - 2 y	0.076	-1.3	0.05	5	ambient	No	No	5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	73.3 % (0.050 and 0.50 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	> 2 y	0.105	-0.3	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		chlormequat chloride D_4	StAdd-SP	80 %	SB-EUPT	2	MeOH extraction
21		Yes	> 2 y	0.114	0.0	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	93 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
23	x	Yes	1 - 2 y	0.12	0.2	0.01	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	93 %	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
25		Yes	> 2 y	0.146	1.1	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	PrCal	107 % (0.05 mg/kg)	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
26	x	No	> 2 y	0.12	0.2		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	86 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	> 2 y	0.1	-0.5	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	111 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
29	x	Yes	> 2 y	0.105	-0.3	0.01	5	slightly frozen	No		mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		No	Centrifugation, Filtration, nylon filter	LC-MS/MS (QQQ), Waters	MM-ML	No	None	102 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.135	0.8	0.005	5	ambient	19.5 mL	after H_2O and organic solvent, 5 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ), 114/98	PS-ML	D_3-mepiquat		94 % (87 % D_3-Mepiqat recovery)	SB-EUPT	1	DIN EN 15055, 2006-08
31		Yes	< 1 y	0.16	1.6	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	106 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
32		No	> 2 y	0.14	0.9	0.01	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.157	1.5																			
34		Yes	> 2 y	0.11	-0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
35		Yes	> 2 y	0.114	0.0	0.005	10	ambient	20 ml	No	mechanical shaking, 15 min	No	Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	mepiquat iodide D_3	None	102 %	SB-EUPT	1	Startin J.R., Hird S.J., Sykes M.D., Taylor J.C. and Hill A.J. Determination of residues...
36		Yes	> 2 y	0.0946	-0.7	0.01	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_3 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.107	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS	MM-ML	No	StAdd-SP	82.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.102	-0.4	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		Yes	> 2 y	0.109	-0.2	0.008	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	PS-ML	D_3-mepiquat					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
44	x	Yes	> 2 y	0.142	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		Yes	> 2 y	0.109	-0.2	0.005	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	102 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.115	0.0	0.005	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	108 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
47	x	Yes	> 2 y	0.106	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	Isooctane				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	75.3 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
49	x	No	> 2 y	1.73	56.9	0.01	5	cold	Yes	after H_2O, 5 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ILIS	None	99 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
51	x	Yes	> 2 y	0.117	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		113 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
54	x	Yes	> 2 y	0.12	0.2	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, Ammonium Acetate	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	StAdd-SP	No	StAdd-SP	75 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 without dilution

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Mepiquat

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
55		Yes	> 2 y	0.126	0.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	115 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
56		Yes	1 - 2 y	0.122	0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/ $MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	36 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		No	1 - 2 y	0.1	-0.5	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	72 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	> 2 y	0.127	0.5	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
59	x	No	< 1 y	0.087	-0.9	0.02	10	cold		after H_2O, 10 min	manual shaking, 2 min	No	Isooctane	No		No	Centrifugation	LC-Ion Trap	PS-ML	ILIS		84 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.11	-0.1	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	104 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.11	-0.1	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	91 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	1 - 2 y	0.173	2.1	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN	Yes (HOAc buffered)			Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	50 %	SB-EUPT		QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
64	x	No	> 2 y	0.116	0.1	0.02	5	deep frozen	10 ml	after H_2O, 10 min		No	H_2O/HCl, Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	104.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.154	1.4	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	73 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
66		Yes	> 2 y	0.122	0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	105 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
67	x	Yes	> 2 y	0.1762	2.2	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.108	-0.2	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	103 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	Yes	> 2 y	0.1	-0.5	0.01	25	cold	Yes	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	D_3-mepiquat	None				MeOH/H_2O extraction
75		Yes	> 2 y	0.103	-0.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	mepiquat D_3	StAdd-EA		QC	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
76		Yes	> 2 y	0.172	2.1	0.01	10	ambient	Yes	after H_2O and organic solvent, 1 min	1 min	No	MeOH	1×				LC-Orbitrap	PS-ML	atrazine	None	117 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Analysis of Dithiocarbamate Residues in Foods of Plant Origin Involving Cleavage into Carbon Disulfide, Partitioning into Isooctane
77		No		0.12	0.2	0.01												no data		No	StAdd-SP				no data
78		Yes	> 2 y	0.071	-1.5	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	69 %	SB-EUPT	1	in house method
80		Yes	> 2 y	0.142	1.0	0.01	10	ambient	10 ml	after H_2O and organic solvent, 10 min	ultra turrax, 1 min	No	ACN	No		No	SPE-column; OASIS WCA	LC-MS/MS (QQQ), Waters Xevo	StAdd-SP	mepiquat D_3	StAdd-SP				other
81	x	Yes	> 2 y	0.137	0.8	0.01	10	cold	19 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	equat chloride D_4	None	115 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
82		Yes	> 2 y	0.136	0.8	0.01	5	ambient	10 ml	No	ultrasonic bath, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
83		No	< 1 y	0.11	-0.1	0.01	10	ambient			mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), using 120 ml of extraction solution
84	x	Yes	> 2 y	0.106	-0.3	0.01	10	slightly frozen	Yes	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	113 %	SB-EUPT	1	EN15055
85		No	< 1 y	0.026	-3.1	0.01	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		60.2 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		Yes	> 2 y	0.126	0.4	0.02	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Centrifugation	LC-MS/MS (QQQ), ESI pos.	MM-ML	mepiquat D_3	None	102.4 %	SB-EUPT	1	§ 64 LFGB, L00.00-76
89		Yes	< 1 y	0.1	-0.5	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min		Isooctane	1× (FA)				LC-MS/MS (QQQ)	MM-ML	TPP	PrCal	98 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
90		Yes	> 2 y	0.0881	-0.9	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane, acidified with FA	1× (by FA within the solvent)		No	Filtration; 0.45 µm	LC-MS/MS (QQQ)	MM-ML	No	None	73 % (0.08 mg/kg)	SB-EUPT	1	§ 64 LFGB, L00.00-76
91	x	No	> 2 y	0.11	-0.1	0.01	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	1 % FA in ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	No	None	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), solvent = 1 % FA in ACN, no ISTD was used
94	x	Yes	> 2 y	0.13	0.6	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	101.9 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.214	3.5		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
97	x	No	> 2 y	0.065	-1.7	0.05	10	ambient	until volume to 20 mL	No	mechanical shaking, 2 min	No	Isooctane, 40 mL MeOH	No		No	No	LC-MS/MS (QQQ), internal standard	MM-SL	mepiquat iodide D_3	None	67 % (0.05 mg/kg)	SB-EUPT	2	analysis of chlormequat and mepiquat residue in foods of plant origin EURLSRM Jan 2009
98	x	Yes	1 - 2 y	0.095	-0.7	0.01	5	deep frozen	Yes	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Centrifugation	LC-MS/MS (QQQ)	MM-ML	No		70 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.48479	13.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	128.07 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
106		Yes	> 2 y	0.11	-0.1	0.01	10	deep frozen	Yes		15 min	No						LC-MS/MS (QQQ)	MM-ML	ILIS	None	93 %	SB-EUPT	1	other (extraction with MeOH)
107		Yes	> 2 y	0.104	-0.3	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	None	75 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
111		Yes	> 2 y	0.09	-0.8	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	chlormequat-D_4	None	89 % (2x MRRL)	SB-EUPT	1	other
113		Yes	> 2 y	0.089	-0.9	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	74 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
114		Yes		0.1	-0.5	0.05														other compound	StAdd-SP	86 %	SB-EUPT	1	no data
116		Yes	> 2 y	0.13	0.6	0.01	2	ambient	Yes	after H_2O and organic solvent, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
118		No	None	0.0154	-3.5		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-SL	chlormequat-D_4	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Propamocarb

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	> 2 y	0.081	0.9	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	47 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.053	-0.8	0.01	10	deep frozen	No	No	ASE, 15 min	No	ACN	No		No	aluminium oxide	LC-MS/MS (QQQ)	StAdd-SP	generic IS	PrCal	101 % (0.05 et 0.1 mg/kg)	SB-EUPT	2	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.098	1.9	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.0532	-0.8	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	propamocarb D_7	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.075	0.5	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.076	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.096	1.8	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		Isooctane			No	No	LC-MS/MS (QQQ)		ILIS	other	78 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
9		Yes	> 2 y	0.087	1.2	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	79 % (not corrected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
10		Yes	> 2 y	0.0787	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	87 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		Yes	> 2 y	0.06	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	pirimicarb-D_6		88 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
12	x	Yes	1 - 2 y	0.068	0.1	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-SP	daminozide D_6	StAdd-SP	91.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.0708	0.3	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	TPP	StAdd-EA	75 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
16		Yes	> 2 y	0.0553	-0.7	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out; $CaCl_2$	LC-MS/MS (QQQ)	PS-ML	No	None	86.3 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
18		Yes	> 2 y	0.081	0.9	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.068	0.1	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	103 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0816	0.9	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		No	StAdd-SP	80 %	SB-EUPT	2	MeOH extraction
21		Yes	None	0.074	0.4	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	94 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
22		Yes	> 2 y	0.0892	1.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered, pH 4; PSA/$MgSO_4$, pH > 8)		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI positiv	MM-ML	pirimicarb-D_6, TDCPP	None	109 % (0.05 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Propamocarb

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
23	x	No	1 - 2 y	0.057	-0.6	0.01	5		10 ml	after H_2O, 30 min	1 min		ACN	No			Freezing out	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	86 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
24	x	No	> 2 y	0.0252	-2.5	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	80 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
25		Yes	> 2 y	0.064	-0.2	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ)	MM-ML	TPP	other	78 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
26	x	No	> 2 y	0.086	1.2		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	86.9 %		3	QuEChERS - Original Version (J. AOAC 86, 2003)
27		Yes	> 2 y	0.072	0.3	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	75 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.055	-0.7	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN, 10 ml	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	91 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003)
30		Yes	> 2 y	0.077	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Centrifu-gation	LC-MS/MS (QQQ), 189/74	MM-ML	No	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
31		No	> 2 y	0.067	0.0	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	120 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
32		Yes	> 2 y	0.056	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.062	-0.3	0.01	5	ambient	5 ml	No	ultra turrax, 2 min	No	MeOH + 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	TPP					in house method
34		Yes	> 2 y	0.02	-2.8	0.02	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	EtOAc, 10 m	No		No	Dessication with Na_2SO_4, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	TPP	None	60 %	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789)
35		No	> 2 y	0.056	-0.6	0.01	10	ambient	20 ml	No	mechanical shaking, 20 min	No	EtOAc	1× ($NaHCO_3$)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	75 % (0.032 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time
36		Yes	> 2 y	0.0523	-0.9	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
37	x	Yes	> 2 y	0.034	-2.0	0.01	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
38		Yes	> 2 y	0.071	0.3	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	81.6 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
40		Yes	< 1 y	0.07	0.2	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
41	x	No	None	0.0769	0.6	0.01	5	ambient	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	70 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		No	> 2 y	0.067	0.0	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	> 2 y	0.0718	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
45		Yes	> 2 y	0.0696	0.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	101 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Propamocarb

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
46		Yes	> 2 y	0.0704	0.2	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	TPP	None	100 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
47	x	Yes	> 2 y	0.057	-0.6	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	77.5 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
49	x	Yes	> 2 y	0.061	-0.3	0.01	5	cold	Yes	after H_2O, min	manual shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-SL	No	None	80 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
51	x	Yes	> 2 y	0.058	-0.5	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	TPP	StAdd-EA	80 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.03	-2.2	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-SL	No	PrCal	86 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.073	0.4	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, Ammonium Acetate	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	94 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66
55		Yes	> 2 y	0.05	-1.0	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	93 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
56		Yes	> 2 y	0.0718	0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/ $MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	45 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.049	-1.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	64 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
58		Yes	> 2 y	0.012	-3.3	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
60	x	No	> 2 y	0.0575	-0.5	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	carbaryl	PrCal	87 %	SB-EUPT	3	Modified QuEChERS
61		Yes	> 2 y	0.08	0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	81 %	QC		QuEChERS-Citrate buffered (EN 15662)
62		Yes	> 2 y	0.062	-0.3	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min		ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No		71 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
63		Yes	> 2 y	0.064	-0.2	0.02	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	40 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
64	x	Yes	> 2 y	0.024	-2.6		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	57.1 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
65	x	No	None	0.0461	-1.2	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	60 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	> 2 y	0.101	2.1	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	ILIS	PrCal	90 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
67	x	Yes	> 2 y	0.1237	3.4	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.0756	0.5	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	100 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
71		No	None	0.115	2.9	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	RecF	61.2 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Propamocarb

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
72		Yes	1 - 2 y	0.0717	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	105 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
73		Yes	> 2 y	0.078	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	104 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.058	-0.5	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No	other				QuEChERS-Citrate buffered (EN 15662)
75		Yes	> 2 y	0.077	0.6	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	StAdd-EA	propamocarb D_7	StAdd-EA	65 %	QC	> 5	QuEChERS-Citrate buffered (EN 15662), additional with QuPPe-Method
76		Yes	> 2 y	0.06	-0.4	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No			Disp.-SPE (PSA/$MgSO_4$)	LC-Orbitrap	PS-ML	atrazine	None	89 %			QuEChERS-Citrate buffered (EN 15662)
77		Yes	> 2 y	0.071	0.3	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
78		Yes	> 2 y	0.065	-0.1	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	ACN	No		No	Centrifugation, Dessication with Na_2SO_4	LC-MS/MS (QQQ)	MM-ML	generic IS	None	83 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003)
79		No	< 1 y	0.092	1.5																				
80		Yes	> 2 y	0.082	0.9	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.0658	0.0	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	88 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
82		Yes	> 2 y	0.072	0.3	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1×		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ), ESI+	MM-ML	No	RecF	29 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662); Unusual low recovery, reason is not clear
83		Yes	> 2 y	0.085	1.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 20 min	No	ACN	1× (buffered pH = 5)		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuEChERS-Citrate buffered (EN 15662)
84	x	Yes	> 2 y	0.055	-0.7	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
85		No	< 1 y	0.067	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		54 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
87		Yes	> 2 y	0.077	0.6	0.01	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Liq-liq part.; ChemElut	LC-MS/MS (QQQ), ESI pos.	MM-ML	carbendazim-D_4	other	93.6 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut; Dilution of extract 1:50
89		Yes	< 1 y	0.052	-0.9	0.005	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	73 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
90		Yes	> 2 y	0.0414	-1.5	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	None	64 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
91	x	Yes	> 2 y	0.0756	0.5	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	81.9 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
94	x	Yes	> 2 y	0.0583	-0.5	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	106 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

COMPULSORY ANALYTES | Propamocarb

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
95		No	< 1 y	0.039	-1.7		5	ambient	10 ml	after H_2O, 15 min	10 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)
97	x	No	> 2 y	0.025	-2.5	0.01	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/$MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	TPP	None	75 % (0.01 mg/kg)	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM, EURL SRM
98	x	Yes	1 - 2 y	0.056	-0.6	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		80 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.08399	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	127.7 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.08	0.8	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			Disp.-SPE; PSA-C18	LC-MS/MS (QQQ)	PS-ML	triclabendazole	RecF	31 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), Purification
105		Yes	> 2 y	0.058	-0.5	0.01	5	ambient	10 ml	after H_2O, 60 min	manual shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	PrCal	80 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), dilution before LC-MS
106		Yes	> 2 y	0.069	0.1	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	81 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
107		Yes	> 2 y	0.0571	-0.6	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
109		Yes	> 2 y	0.06	-0.4	0.01	2	slightly frozen	10 ml	No	manual shaking, 2 min		ACN	No			Freezing out, Disp.-SPE (PSA/$MgSO_4$)	GC-MSD	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)
111		Yes	> 2 y	0.06	-0.4	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	TBP	None	91 % (2x MRRL)	SB-EUPT	1	other
113		No	> 2 y	0.055	-0.7	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	104 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
114		Yes		0.027	0.4	0.01														generic IS	StAdd-SP	96 %	SB-EUPT	1	no data
116		Yes	> 2 y	0.15	5.0	0.02	2	ambient	No	No	mechanical shaking, 5 min	No	ACN, H_2O	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	102 %	SB-EUPT	1	other (extraction with ACN:H_2O)
118		No	None	0.0884	1.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	TPP	None			4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.1	0.1	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column	LC-MS/MS (QQQ)	StAdd-SP	MCPP-D_3	PrCal	92 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.104	0.3	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	110 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.0917	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.12	0.9	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.094	-0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.082	-0.6	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	87 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
10		Yes	> 2 y	0.1	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		No	< 1 y	0.099	0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		101 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
12	x	No	None	0.118	0.8	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	109.5 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
15		Yes	> 2 y	0.0958	-0.1	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	83 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
16		Yes	> 2 y	0.071	-1.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
18		Yes	> 2 y	0.073	-1.0	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.1	0.1	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.075	-0.9	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	70 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		Yes	> 2 y	0.11	0.5	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	117 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
24	x	No	> 2 y	0.0338	-2.6	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	89 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
25		Yes	1 - 2 y	0.103	0.2	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	114 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
26	x	No	1 - 2 y	0.101	0.1		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	76.8 %		3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.16	2.5	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	93 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
28		No	> 2 y	0.1	0.1	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	99 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
30		Yes	> 2 y	0.084	-0.6	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabu-tyl-ammoni-umhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:112/154/133	PS-SL	No	None	85 %	SB-EUPT	1	other (with methylation)
31		No	1 - 2 y	0.07	-1.1	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	78 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
32		No	None	0.101	0.1	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.126	1.2	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
34		Yes	> 2 y	0.049	-2.0	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	No	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	111 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
36		Yes	> 2 y	0.0754	-0.9	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
38		Yes	> 2 y	0.092	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	95.8 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
40		Yes	> 2 y	0.093	-0.2	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifu-gation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	84 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.109	0.5	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	1 - 2 y	0.115	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
45		Yes	> 2 y	0.107	0.4	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	99 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
46		Yes	1 - 2 y	0.112	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	bentazone D_7	None	99 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.105	0.3	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	ILIS	StAdd-EA	113 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.1	0.1	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	101 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.105	0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, Am-monium Acetate	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	93 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66
55		Yes	> 2 y	0.102	0.2	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	bentazone-D_6	PrCal	94 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
56		Yes	> 2 y	0.0947	-0.1	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	101 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.116	0.7	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	119 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
58		Yes	> 2 y	0.068	-1.2	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
60	x	No	< 1 y	0.134	1.5																				
61		Yes	> 2 y	0.097	0.0	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	98 %	QC		QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.14	1.7	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	50 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
64	x	Yes	> 2 y	0.038	-2.4	0.05	15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	31.9 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
65	x	No	< 1 y	0.0898	-0.3	0.02	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	68 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	1 - 2 y	0.0893	-0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	100 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.0834	-0.6	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		90 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
70	x	No	None	0.0806	-0.7	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	95 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
71		No	None	0.083	-0.6	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	73.7 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
72		Yes	1 - 2 y	0.105	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	89 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
73		No	None	0.0989	0.0	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	100.7 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.103	0.2	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
75		Yes	> 2 y	0.12	0.9	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	109 %	QC	> 5	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.125	1.1	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
78		Yes	> 2 y	0.092	-0.2	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	83 %	SB-EUPT	1	in house method
79		No	< 1 y	0.096	-0.1	0.01	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	84 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
80		Yes	> 2 y	0.103	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
82		Yes	> 2 y	0.116	0.7	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	59 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.102	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
84	x	No	None	0.093	-0.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
85		No	< 1 y	0.044	-2.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		26 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
87		Yes	> 2 y	0.09	-0.3	0.01	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	77.8 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
89		Yes	< 1 y	0.094	-0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
91	x	No	< 1 y	0.0921	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	93.7 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
95		No	< 1 y	0.106	0.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)
98	x	Yes	1 - 2 y	0.068	-1.2	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		93 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.17459	3.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	130.05 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		No	None	0.081	-0.7	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	No	None	79 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
106		Yes	> 2 y	0.11	0.5	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	102 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
107		Yes	> 2 y	0.101	0.1	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	0.116	0.7	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	95 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
114		No		0.029	-2.8	0.01														generic IS	StAdd-SP	100 %	SB-EUPT	1	no data
115		Yes	None	0.02	-3.2		10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
116		No	None	0.09	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
117		Yes	1 - 2 y	0.097	0.0	0.01	2	just thawed	Yes	after H_2O, min	manual shaking		ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	97 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
118		No	None	0.0308	-2.7		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	TPP	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No	> 2 y	0.155	1.0	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	None	112 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.114	-0.4	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column	LC-MS/MS (QQQ)	StAdd-SP	MCPP-D_3	PrCal	117 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.125	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.133	0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.147	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.139	0.4	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.111	-0.5	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	95 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM
9		Yes	1 - 2 y	0.137	0.4	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	20 % NaOH; pH 12	Isooctane	1× (3-4)			SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	80 % (not corrected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
10		Yes	> 2 y	0.133	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	97 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		Yes	> 2 y	0.121	-0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		95 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
12	x	Yes	1 - 2 y	0.13	0.2	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	102.3 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
15		Yes	> 2 y	0.123	-0.1	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	87 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
18		Yes	> 2 y	0.083	-1.3	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.15	0.8	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	115 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0805	-1.4	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	103 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		Yes	> 2 y	0.127	0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
22		Yes	> 2 y	0.129	0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4), PSA/$MgSO_4$ (pH > 8))		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	99 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
23	x	Yes	1 - 2 y	0.146	0.7	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	113 %	SB-EUPT	3	other

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
24	x	No	> 2 y	0.0579	-2.2	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
25		Yes	> 2 y	0.151	0.8	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	128 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
26	x	No	1 - 2 y	0.147	0.7		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77.3 %		3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.148	0.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	102 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
28		No	1 - 2 y	0.146	0.7	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	86 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.13	0.2	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	84 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
30		Yes	> 2 y	0.1	-0.8	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabu-tyl-ammoni-umhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:91/276/289	PS-SL	No	None	91 %	SB-EUPT	1	other (with methylation)
31		No	1 - 2 y	0.14	0.5	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	80 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
32		No	None	0.105	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.154	0.9	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
34		Yes	> 2 y	0.039	-2.8	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	No	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	71 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
36		Yes	> 2 y	0.0636	-2.0	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
40		Yes	> 2 y	0.118	-0.2	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifu-gation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.132	0.2	0.003	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
45		Yes	> 2 y	0.127	0.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	96 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
46		Yes	1 - 2 y	0.127	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	94 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.127	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	111 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.118	-0.2	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
55		Yes	> 2 y	0.138	0.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	bentazone-D_6	PrCal	104 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
56		Yes	> 2 y	0.12	-0.2	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	102 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.116	-0.3	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
58		No	> 2 y	0.091	-1.1	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
60	x	No	> 2 y	0.173	1.5																				
61		Yes	> 2 y	0.1	-0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	99 %	QC		QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.13	0.2	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	45 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
64	x	Yes	> 2 y	0.062	-2.0		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	39.6 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
66		Yes	1 - 2 y	0.126	0.0	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	100 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.1161	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		78 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
70	x	No	None	0.124	0.0	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	60 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
72		Yes	1 - 2 y	0.135	0.3	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
73		No	None	0.116	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	90 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
74	x	No	None	0.146	0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
75		Yes	> 2 y	0.12	-0.2	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	97 %	QC	> 5	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.128	0.1	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
78		Yes	> 2 y	0.099	-0.8	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	81 %	SB-EUPT	2	in house method
80		Yes	> 2 y	0.113	-0.4	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.12	-0.2	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	93 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
82		Yes	> 2 y	0.153	0.9	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	66 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.132	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
84	x	Yes	> 2 y	0.122	-0.1	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	91 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
85		No	< 1 y	0.08	-1.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		102 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
87		Yes	> 2 y	0.108	-0.6	0.02	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	66.9 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
89		Yes	< 1 y	0.115	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	60 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
91	x	Yes	> 2 y	0.124	0.0	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.6 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.20976	2.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	135.22 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.104	-0.7	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	No	None	90 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
106		Yes	> 2 y	0.14	0.5	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	100 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
113		No			-3.7 (FN)		5	ambient										LC-MS/MS (QQQ)							A-QuEChERS (with 1 % FA)
114		Yes		0.043	-2.6	0.02														generic IS	StAdd-SP	98 %	SB-EUPT	1	no data
115		Yes			-3.7 (FN)	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML						QuEChERS-Citrate buffered (EN 15662), modified method
117		Yes	1 - 2 y	0.108	-0.6	0.01	2	just thawed	Yes	after H_2O, min			ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	113 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
118		No	None	0.0521	-2.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

OPTIONAL ANALYTES | N-Acetyl glufosinate

N-Acetyl glufosinate (Assigned value = 0.319 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.329	0.1	0.02	3	deep frozen	9.75 mL	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	PS-ML	No		101 % (0.16 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Cf. extraction, clean up and chromatography
4	x	Yes	1 - 2 y	0.337	0.2	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	glufosinate D_3	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	No	< 1 y	0.305	-0.2	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
12	x	Yes	> 2 y	0.279	-0.5	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	N-acetyl glufosinate D_3	StAdd-EA	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.278	-0.5	0.02	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	83 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		No	None	0.321	0.0	0.02	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	No	RecF	93 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
18		No	1 - 2 y	0.35	0.4	0.02	5	ambient	10 ml.		20 min		Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.318	0.0	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		glufosinate D_3	StAdd-SP	90 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
21		No	None	0.335	0.2	0.02	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	91 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
25		Yes	< 1 y	0.306	-0.2	0.05	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	PrCal	83 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
51	x	No	< 1 y	0.416	1.2	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	glyphosate 2-^{13}C	StAdd-EA	113 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	None	0.356	0.5	0.1	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
60	x	No			-3.7 (FN)	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			Centrifugation	no data	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	1 - 2 y	0.35	0.4	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	101 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	No	None	0.26	-0.7	0.05	5	cold	Yes	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	No	> 2 y	0.282	-0.5	0.02	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			Centrifugation	LC-MS/MS (QQQ)	MM-ML	N-acetyl glufosinate-D_3 disodium salt	None	86 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Phosphonic acid (Assigned value = 0.584 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
4	x	Yes	< 1 y	0.615	0.2	0.05	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	phosphonic acid $^{18}O_3$	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.971	2.6	0.05	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	No	< 1 y	0.598	0.1	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides); no ISTD used for reported result
8		No	1 - 2 y	0.553	-0.2	0.1	1	ambient	9 ml	after H_2O and organic solvent, 5 min	ultra turrax		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)		ILIS	other	89 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM
10		Yes	< 1 y	0.617	0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	$^{18}O_4$ phosphonic acid	None	95 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No			-3.7 (FN)	0.05	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.585	0.0	0.1	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		phosphonic acid $^{18}O_3$	StAdd-SP	94 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
21		Yes	1 - 2 y	0.559	-0.2	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	82 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
25		Yes	1 - 2 y	0.334	-1.7	0.1	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	118 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	None	0.606	0.1	0.1	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	No	StAdd-SP	73 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.591	0.0	0.1	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ), 81/79	PS-ML	$^{18}O_3$ phosphonic acid	PrCal	80 % (83 % O_3 phosphonic acid recovery)	SB-EUPT	1	in house method, QuPPe-based
33		Yes	1 - 2 y	0.774	1.3	0.05	5	ambient	5 ml	No	mechanical shaking, 30 min	No	H_2O, MeOH 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	phosphonic acid $^{18}O_3$					in house method
46		No	None	0.55	-0.2	0.05	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	phosphonic acid $^{18}O_3$	None	98 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
53		No	> 2 y	0.469	-0.8	0.05	5	deep frozen	10 ml		manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)			No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	74 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
57		No	1 - 2 y	0.276	-2.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	None	0.592	0.1	0.05	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
60	x	No	1 - 2 y	0.226	-2.5	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	75 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	1 - 2 y	0.56	-0.2	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	111 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	1.106	3.6	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	PrCal	60 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

Phosphonic acid (Assigned value = 0.584 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
70	x	No	None	0.672	0.6	0.1	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	ILIS	None	88 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
78		Yes	< 1 y	0.76	1.2		10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	1×		No	Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS	None	114 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	No	None	0.717	0.9	0.05	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	phosphonic acid $^{18}O_3$	None	100 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	1 - 2 y	0.566	-0.1	0.1	5	ambient	10 ml	No	manual shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		No	< 1 y	0.534	-0.3	0.3	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	phosphonic acid $^{18}O_3$	other	93.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:20
100		Yes	> 2 y	0.35534	-1.6	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	139.06 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

TFNG (Assigned value = 0.168 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
3		Yes	> 2 y	0.174	0.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.197	0.7	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.217	1.2	0.02	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	No	< 1 y	0.208	1.0	0.02	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
10		No	> 2 y	0.159	-0.2	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
12	x	No	None	0.132	-0.8	0.02	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	86.5 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
20		Yes	> 2 y	0.17	0.1	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	101 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		No	None	0.164	-0.1	0.02	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	84 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
25		Yes	< 1 y	0.174	0.2	0.05	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN, 1 % FA	No				LC-MS/MS (QQQ)	MM-ML	nicarbazin	other	93 % (0.05 mg/kg)	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
27		Yes	> 2 y	0.186	0.4	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	96 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	< 1 y	0.165	-0.1	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	75 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
30		Yes	> 2 y	0.14	-0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$), Centrifu-gation	LC-MS/MS (QQQ), 247/182	MM-ML	No		78 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
33		Yes	1 - 2 y	0.18	0.3	0.02	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	TPP					QuEChERS - Original Version (J. AOAC 86, 2003)
34		No	None	0.029	-3.3	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Disp.-SPE (PSA/ $MgSO_4$), Centrifu-gation	LC-MS/MS (QQQ), ESI pos	MM-ML	TPP	None	95 %	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
45		No	1 - 2 y	0.162	-0.1	0.02	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	95 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
51	x	Yes	1 - 2 y	0.164	-0.1	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	119 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
53		No	< 1 y	0.212	1.1	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	109 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
55		Yes	1 - 2 y	0.18	0.3	0.02	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	92 % (0.04 mg/kg)	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
58		No	None	0.014	-3.7	0.02	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
61		Yes	1 - 2 y	0.2	0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	88 %	QC		QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes)

TFNG (Assigned value = 0.168 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
67	x	No	None	0.16	-0.2	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		79 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
72		Yes	1 - 2 y	0.165	-0.1	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	111 %	SB-EUPT	3	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
73		No	None	0.14	-0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	78.8 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.16	-0.2	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
81	x	Yes	< 1 y	0.134	-0.8	0.02	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	69 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
82		Yes	1 - 2 y	0.21	1.0	0.05	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	40 % (0.1 mg/kg)	SB-other	4	QuEChERS-Citrate buffered (EN 15662); Unusal low recovery, reason is not clear
87		No	< 1 y	0.102	-1.6	0.04	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	83 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:50
91	x	No	< 1 y	0.188	0.5	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	94.2 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.19125	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	122.41 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
103		Yes	< 1 y	0.156	-0.3	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	50 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
113		Yes	> 2 y	0.134	-0.8	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	85 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

COMPULSORY ANALYTES | 2,4-D (free acid)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
24	x	No	> 2 y	0.0131	-3.4	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
34		Yes	> 2 y	0.026	-2.9	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Centrifugation	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	76 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
114		No		0.028	-2.8	0.01														other compound	StAdd-SP	104 %	SB-EUPT	1	no data
115		Yes	None	0.03	-2.7	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
58		Yes	> 2 y	0.049	-1.9	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
79		No	< 1 y	0.055	-1.6	0.05	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
78		Yes	> 2 y	0.058	-1.5	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	62 %	SB-EUPT	2	in house method
97	x	Yes	> 2 y	0.06	-1.4	0.02	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	nicarbazin	None	75 % (0.02 mg/kg)	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM, EURL SRM
31		Yes	< 1 y	0.062	-1.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	120 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
16		Yes	> 2 y	0.065	-1.2	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	63.5 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
18		Yes	> 2 y	0.068	-1.0	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
35		Yes	> 2 y	0.0706	-0.9	0.01	5	ambient	10 ml	No	mechanical shaking, 30 min	No	EtOAc with 1 % HOAc	1× (1 % HOAc)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	72 % (0.101 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time
59	x	Yes	> 2 y	0.072	-0.9	0.05	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min	NaOH	ACN	1× (H_2SO_4)		Yes	C18/$MgSO_4$	GC-MSD (following derivatization)	MM-ML	No	None	77 %	SB-EUPT	2	other (with derivatization)
65	x	Yes	> 2 y	0.0735	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	70 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0767	-0.7	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	71 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
74	x	Yes	> 2 y	0.076	-0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
85		No	> 2 y	0.076	-0.7	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		62 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
28		No	> 2 y	0.078	-0.6	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	90 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.081	-0.5	0.005	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA				QuEChERS-Citrate buffered (EN 15662)
76		Yes	> 2 y	0.08	-0.5	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No				LC-Orbitrap	PS-ML	2.4,6-trichloro-phenol	None	93 %			A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.082	-0.4	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML	MCPP-D_3	PrCal	112 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
32		Yes	> 2 y	0.082	-0.4	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
38		Yes	> 2 y	0.083	-0.4	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
60	x	No	> 2 y	0.0829	-0.4	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	103 %	SB-EUPT	3	Modified QuEChERS
64	x	Yes	> 2 y	0.083	-0.4		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	other	28.9 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step; Sample corrected for QC recovery
81	x	Yes	> 2 y	0.084	-0.4	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
91	x	Yes	> 2 y	0.0826	-0.4	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.4 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
107		Yes	> 2 y	0.082	-0.4	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
56		Yes	> 2 y	0.0854	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	98 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
73		No	None	0.0849	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	93.5 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.086	-0.3	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
83		Yes	> 2 y	0.086	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
118		No	None	0.0853	-0.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
21		Yes	> 2 y	0.088	-0.2	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
51	x	Yes	> 2 y	0.088	-0.2	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	StAdd-EA	nicarbazin	StAdd-EA	101 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
84	x	Yes	> 2 y	0.087	-0.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
90		Yes	> 2 y	0.0875	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	91 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
8		Yes	> 2 y	0.09	-0.1	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	86 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
61		Yes	> 2 y	0.09	-0.1	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	96 %	QC		QuEChERS-Citrate buffered (EN 15662)
70	x	Yes	> 2 y	0.0906	-0.1	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	62 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
9		Yes	1 - 2 y	0.092	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	20 % NaOH; pH 12	Isooctane	1× (3-4)			SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	83 % (not corrected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
15		Yes	> 2 y	0.0923	0	0.004	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	95 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
26	x	No	> 2 y	0.092	0		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	64.7 %		3	QuEChERS-Citrate buffered (EN 15662)
40		Yes	> 2 y	0.092	0	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifugation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	80 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.092	0	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	99 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 with extract conc
55		Yes	> 2 y	0.092	0	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	(3-chloro-4-methyl-phenoxy)-HOAc	PrCal	129 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
57		Yes	> 2 y	0.092	0	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	113 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.093	0	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	69 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
1		Yes	> 2 y	0.095	0.1	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	97 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
5		Yes	> 2 y	0.095	0.1	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	2.5-dichlorobenzoic acid	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.094	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	103 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
37	x	Yes	> 2 y	0.095	0.1	0.05	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	108 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA cleaning
87		Yes	> 2 y	0.095	0.1	0.02	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	67.3 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
11		Yes	> 2 y	0.096	0.2	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		101 % (0.02 and 0.1 mg/kg)	SB-EUPT	3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
3		Yes	> 2 y	0.098	0.3	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	115 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.098	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
12	x	Yes	1 - 2 y	0.098	0.3	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	100.4 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
44	x	Yes	> 2 y	0.1	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
45		Yes	> 2 y	0.0986	0.3	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	92 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
53		No	> 2 y	0.1	0.3	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	122 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	> 2 y	0.1	0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
72		Yes	> 2 y	0.0982	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
98	x	Yes	1 - 2 y	0.102	0.4	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE ($PSA/MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		91 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
22		Yes	> 2 y	0.103	0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4)		No	Disp.-SPE ($PSA/MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	97 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
75		Yes	> 2 y	0.104	0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA	84 %	QC	> 5	A-QuEChERS (with 1 % FA)
89		Yes	< 1 y	0.104	0.5	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	76 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
117		Yes	1 - 2 y	0.104	0.5	0.01	2	just thawed	Yes	H_2O,	manual shaking		ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	91 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
10		Yes	> 2 y	0.106	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
25		Yes	> 2 y	0.106	0.6	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	95 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
30		Yes	> 2 y	0.105	0.6	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabutyl-ammoniumhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:401/400/391	PS-SL	No	None	88 %	SB-EUPT	1	alkaline hydrolysis extraction, GPC, acid/base distribution, methylation, GC-MSD detection
80		Yes	> 2 y	0.105	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
36		Yes	> 2 y	0.109	0.7	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	RecF	61 % (Avegrage from ongoing AQC samples)	QC	> 5	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
46		Yes	1 - 2 y	0.108	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	95 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
19		Yes	1 - 2 y	0.11	0.8	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93.5 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.111	0.8	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE ($PSA/MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	107 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
67	x	Yes	> 2 y	0.1106	0.8	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		95 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

COMPULSORY ANALYTES | 2,4-D (free acid)

2,4-D (free acid) (Assigned value = 0.092 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1)]	ISTD used [2)]	Other means of correcting for recovery or matrix-effects? [3)]	Recovery % (compound. level)	Recovery obtained from [4)]	Recovery replicates considered	Method details / Comments
94	x	Yes	> 2 y	0.111	0.8	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	105.9 % (0.01 mg/Kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
106		Yes	> 2 y	0.11	0.8	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	91 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
116		Yes	> 2 y	0.11	0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
82		Yes	> 2 y	0.112	0.9	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	None			4	QuEChERS-Citrate buffered (EN 15662)
23	x	Yes	1 - 2 y	0.118	1.1	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	110 %	SB-EUPT	3	other
47	x	No	None	0.123	1.3	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	81.8 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
113		Yes	> 2 y	0.125	1.4	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	110 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
4	x	No	1 - 2 y	0.127	1.5	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	2,4-D D_3	StAdd-SP			1	A-QuEChERS (with 1 % FA)
63		Yes	1 - 2 y	0.13	1.6	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	55 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
71		No	None	0.133	1.8	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87.4 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
33		Yes	> 2 y	0.135	1.9	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
100		Yes	> 2 y	0.14365	2.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	122.19 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
118		No	None	0.00094	-4.0		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-SL	chlormequat-D_4	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
115		Yes	None	0.02	-3.5	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
107		Yes	> 2 y	0.0614	-2.5	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	None	79 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
85		No	< 1 y	0.065	-2.4	0.01	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		67.2 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
36		Yes	> 2 y	0.0804	-2.1	0.01	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_4 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
97	x	No	> 2 y	0.09	-1.8	0.05	10	ambient	until volume to 20 mL	No	mechanical shaking, 2 min	No	Isooctane, 40 mL MeOH	No		No	No	LC-MS/MS (QQQ), internal standard	MM-SL	chlormequat-D_4	None	62 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), analysis of chlormequat and mepiquat residue in foods of plant origin EURLSRM Jan 2009
78		Yes	> 2 y	0.1	-1.6	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	68 %	SB-EUPT	1	in house method
57		No	1 - 2 y	0.117	-1.2	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
34		Yes	> 2 y	0.12	-1.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	80 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
116		Yes	> 2 y	0.12	-1.1	0.01	2	ambient	Yes	after H_2O and organic solvent, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
19		Yes	1 - 2 y	0.13	-0.9	0.05	5	ambient	No	No	5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	88.6 % (0.050 and 0.50 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	Yes	> 2 y	0.136	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
16		Yes	< 1 y	0.136	-0.8	0.01	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	chlormequat chloride D_4	None	82.4 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.134	-0.8	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides); Dilution of extract 1:50
3		Yes	> 2 y	0.137	-0.7	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.152	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	81 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		Yes	> 2 y	0.15	-0.4	0.005	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	91 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.15	-0.4	0.01	5	cold	No	No	mechanical shaking, 10 min	No	Isooctane	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	98 %	QC		Shaking with MeOH

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

COMPULSORY ANALYTES | Chlormequat

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.155	-0.3	0.01	10	deep frozen	No	No	mechanical shaking, 60 min	No	H_2O / MeOH / HCl	No		No	Disp.-SPE; alumina	LC-MS/MS (QQQ)	PS-ML	chlormequat-D_4	None	91 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
18		Yes	> 2 y	0.154	-0.3	0.01	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
29	x	Yes	> 2 y	0.154	-0.3	0.01	5	slightly frozen	No		mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		No	Centrifugation, Filtration, nylon filter	LC-MS/MS (QQQ), Waters	MM-ML	chlormequat-D_4	None	91 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.154	-0.3	0.01	5	ambient	5 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	chlormequat-D_4					in house method
43		Yes	> 2 y	0.155	-0.3	0.008	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	PS-ML	D_4-chlormequat					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
54	x	Yes	> 2 y	0.153	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	StAdd-SP	No	StAdd-SP	69 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 without dilution
56		Yes	1 - 2 y	0.156	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/ $MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	25 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
67	x	Yes	> 2 y	0.1542	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.159	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	chlormequat-D_4	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.159	-0.2	0.01	10	deep frozen	10 ml	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ)	MM-ML	D_4-chlorme-quatchlorid		96 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	other
15		Yes	> 2 y	0.158	-0.2	0.004	5	ambient	No	after H_2O and organic solvent, 10 min	mechanical shaking, 20 min	No		1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	84 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), extraction mixture: 20 mL 80 % ACN in H_2O, 1 % FA
35		Yes	> 2 y	0.161	-0.2	0.005	10	ambient	20 ml	No	mechanical shaking, 15 min	No	Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	chlormequat chloride D_4	None	95 %	SB-EUPT	1	Startin J.R., Hird S.J., Sykes M.D., Taylor J.C. and Hill A.J. Determination of residues...
83		Yes	> 2 y	0.158	-0.2	0.01	10	ambient			mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), using 120 ml of extraction solution
38		Yes	> 2 y	0.162	-0.1	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS	MM-ML	No	StAdd-SP	61.1 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
59	x	No	< 1 y	0.163	-0.1	0.02	10	cold	20 ml	after H_2O, 10 min	manual shaking, 2 min	No	Isooctane	No		No	Centrifugation	LC-Ion Trap	PS-ML	ILIS		110 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
84	x	Yes	> 2 y	0.163	-0.1	0.01	10	slightly frozen	Yes	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	137 %	SB-EUPT	1	EN15055
90		Yes	> 2 y	0.165	-0.1	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane, acidified with FA	1× (by FA within the solvent)		No	Filtration; 0.45 µm	LC-MS/MS (QQQ)	MM-ML	No	None	85 % (0.08 mg/kg)	SB-EUPT	1	§ 64 LFGB, L00.00-76
111		Yes	> 2 y	0.162	-0.1	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	chlormequat-D_4	None	93 % (2x MRRL)	SB-EUPT	1	other
9		Yes	> 2 y	0.167	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				Filtration	LC-MS/MS (QQQ)	PS-ML	isotope D_4	PrCal	103 % (not cor-rected)	SB-EUPT	3	SIST EN 15055:2006
12	x	Yes	> 2 y	0.168	0.0	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	StAdd-EA	98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
47	x	Yes	> 2 y	0.168	0.0	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	78.8 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
51	x	Yes	> 2 y	0.169	0.0	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		109 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.166	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	97 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
64	x	No	> 2 y	0.168	0.0	0.02	5	deep frozen	10 ml	after H_2O, 10 min		No	H_2O/HCl, Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95.1 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.167	0.0	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	102 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.167	0.0	0.01	5	ambient	10 ml	No	ultrasonic bath, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
94	x	Yes	> 2 y	0.166	0.0	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93.4 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	> 2 y	0.172	0.1	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		chlormequat chloride D_4	StAdd-SP	74 %	SB-EUPT	2	MeOH extraction
21		Yes	> 2 y	0.171	0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	89 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
23	x	Yes	1 - 2 y	0.171	0.1	0.01	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	101 %	SB-EUPT	3	other (EN 15055, 2006-08)
27		Yes	> 2 y	0.17	0.1	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	104 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	Yes	> 2 y	0.17	0.1	0.01	25	cold	Yes	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	D_4-chlormequat	None				MeOH/H_2O extraction
75		Yes	> 2 y	0.17	0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	chlormequat-D_4	StAdd-EA		QC	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	Yes	> 2 y	0.171	0.1	0.01	10	cold	19 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	chlormequat chloride D_4	None	107 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		Yes	> 2 y	0.172	0.1	0.015	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Centrifugation	LC-MS/MS (QQQ), ESI pos.	MM-ML	CCC-D_4	None	84.9 %	SB-EUPT	1	§ 64 LFGB, L00.00-76
98	x	Yes	1 - 2 y	0.172	0.1	0.01	5	deep frozen	Yes	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Centrifugation	LC-MS/MS (QQQ)	MM-ML	No		62 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.179	0.3	0.005	5	ambient	19.5 mL	after H_2O and organic solvent, 5 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ), 122/58	PS-ML	D_4-chlormequat	None	93 % (84 % D_4-CCC recovery)	SB-EUPT	1	DIN EN 15055, 2006-08
46		Yes	1 - 2 y	0.178	0.3	0.005	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	None	99 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.18	0.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
26	x	No	> 2 y	0.183	0.4		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	85.4 %		3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
66		Yes	> 2 y	0.185	0.4	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	102 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

COMPULSORY ANALYTES | Chlormequat

Chlormequat (Assigned value = 0.167 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
91	x	No	> 2 y	0.183	0.4	0.01	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	1 % FA in ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	No	None	95.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), solvent = 1 % FA in ACN, no ISTD was used
58		No	> 2 y	0.194	0.6	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
63		Yes	< 1 y	0.194	0.6	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN	Yes (HOAc buffered)			Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	50 %	SB-EUPT		QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
113		Yes	> 2 y	0.192	0.6	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	None	90 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
44	x	Yes	> 2 y	0.198	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
80		Yes	> 2 y	0.197	0.7	0.01	10	ambient	10 ml	after H_2O and organic solvent, 10 min	ultra turrax, 1 min	No	ACN	No		No	SPE-column; OASIS WCA	LC-MS/MS (QQQ), Waters Xevo	StAdd-SP	chlormequat-D_4	StAdd-SP				other
89		Yes	< 1 y	0.197	0.7	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min		Isooctane	1× (FA)				LC-MS/MS (QQQ)	MM-ML	TPP	PrCal	120 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.2	0.8	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
1		No	> 2 y	0.208	1.0	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	31 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
106		Yes	> 2 y	0.21	1.0	0.01	10	deep frozen	Yes		15 min	No						LC-MS/MS (QQQ)	MM-ML	ILIS	None	91 %	SB-EUPT	1	other (extraction with MeOH)
10		Yes	> 2 y	0.212	1.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	None	96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	> 2 y	0.214	1.1	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	chlormequat-D_4	PrCal	102 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
8		No	> 2 y	0.226	1.4	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		Isooctane				No	LC-MS/MS (QQQ)		ILIS	other	93 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
32		No	> 2 y	0.24	1.7	0.01	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.249	1.9	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	75 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
77		No		0.245	1.9	0.01												no data		No	StAdd-SP				no data
55		Yes	> 2 y	0.258	2.2	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	93 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
31		Yes	> 2 y	0.28	2.7	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	114 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.6009	10.4	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	146.64 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
49	x	No	> 2 y	1.35	28.3	0.01	5	cold	Yes	after H_2O, 5 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ILIS	None	100 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
95		No			-3.6 (FN)	0.05	50	ambient	No		> 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MS/MS (QQQ)	PS-ML						$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
100		Yes	> 2 y	0.05622	-3.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	$SnCl_2$/HCl	ACN	HCl		No	No	GC-MS/MS (QQQ)	MM-ML	CS_2-^{13}C	None				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
109		Yes	> 2 y	0.196	-2.6	0.01	5	ambient	10 ml	No	ultrasonic bath, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl				GC-(μ) ECD	MM-ML	No					$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
23	x	Yes	1 - 2 y	0.21	-2.5	0.05	200		No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No	None	76 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
30		Yes	> 2 y	0.228	-2.4	0.01	100	ambient	200 mL		60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer, 272 nm	PS-ML	No	None	98 % (0.15 mg/kg spiking lvel)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
27		Yes	> 2 y	0.27	-2.1	0.02	2	ambient	Yes	No	mechanical shaking, 15 min	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MSD	StAdd-SP	other compound	StAdd-SP		SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
37	x	Yes	> 2 y	0.28	-2.0	0.01	13	ambient	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD, SIM	PS-ML		None	96 % (Hydrolysis recovery with added Thiram)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
94	x	Yes	> 2 y	0.292	-1.9	0.01	25	deep frozen	25 g	No		$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	No	None	40.8 % (0.5 mg/kg (with maneb))	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2, GC-MS used (m/z 76)
5		Yes	> 2 y	0.32	-1.7	0.05	5	ambient	No	No		$SnCl_2$/HCl	H_2O/HCl	HCl		$SnCl_2$/HCl	No	GC-MSD	StAdd-SP	thiophene	PrCal	95 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
26	x	No	> 2 y	0.325	-1.7		50	ambient	45 mL	after H_2O, 15 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-(P) FPD	PS-ML	No		86.7 %		3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
58		Yes	> 2 y	0.328	-1.7	0.05	2	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No		GC-MSD	StAdd-SP	thiophene	StAdd-SP			3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
41	x	No	None	0.334	-1.6	0.05	4	ambient	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-(S) FPD	PS-ML	No	StAdd-SP	75 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
12	x	No	None	0.392	-1.2	0.05	2	cold	2 ml	No	mechanical shaking, 1 min	$SnCl_2$/HCl, 1 h, 80 °C	H_2O/HCl	HCl		No	No	GC-MSD	StAdd-SP	No	StAdd-SP				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
44	x	Yes	> 2 y	0.393	-1.2	0.05	25	ambient	40 ml	after H_2O, 5 min	ultrasonic bath, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD		No	StAdd-SP				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
117		Yes	> 2 y	0.392	-1.2	0.01	25	just thawed	Yes	after H_2O, min	manual shaking	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	68 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
90		Yes	1 - 2 y	0.423	-1.0	0.05	10	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	Dichlormethane	None	63 % (0.5 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
15		Yes	> 2 y	0.435	-0.9	0.0005		cold	No		mechanical shaking	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-Ion Trap, standard addition	StAdd-SP	No	StAdd-SP	108 % (0.2 mg/kg)	SB-EUPT	4	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type), weight 0.5 g of sample
54	x	Yes	> 2 y	0.432	-0.9	0.125	10	ambient	15 ml	after H_2O, 5 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD, Agilent 7000c QQQ	PS-ML	No	None	57 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, isooctane
8		No	> 2 y	0.45	-0.8	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	manual shaking	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MS/MS (QQQ)		No	other	86 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
56		Yes	> 2 y	0.451	-0.8	0.04	50					$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	102 % (Spiking at LOQ)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
57		No	> 2 y	0.452	-0.8	0.01	5	ambient	Yes	No		$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-MSD	MM-ML	chloroform	None	101 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
93		Yes	> 2 y	0.45	-0.8	0.05	100		200 mL	after H_2O, 10 min		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	83.4 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
6	x	Yes	> 2 y	0.458	-0.7	0.04	25	ambient				$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	MM-ML	DCM	PrCal	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
10		Yes	> 2 y	0.462	-0.7	0.05	25	ambient	25 ml	after H_2O and organic solvent, 5 min	mechanical shaking, > 60 min	$SnCl_2$/HCl, release of CS_2	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)		No	No	GC-MSD	PS-ML	No	None	89 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, GC-MS detection
31		Yes	> 2 y	0.46	-0.7	0.01	3	ambient	3 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	HS-CG-MS	PS-ML	No		62.5 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
50		Yes	> 2 y	0.465	-0.7	0.05	25	slightly frozen	Yes	after H_2O and	manual shaking,	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-	HCl		No		GC-(P) FPD	PS-ML	No	None	76 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-
75		Yes	> 2 y	0.459	-0.7	0.05	25	ambient	see Solvent 1	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Distillation passing through NaOH+ H_2SO_4	Spectrophotometer	PS-ML	No	None	87 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
108		Yes	> 2 y	0.47	-0.6							$SnCl_2$/HCl	H_2O/HCl	HCl				no data		No		83 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
112		No	None	0.47	-0.6	0.1	50	slightly frozen	45 mL	after H_2O, 5 min	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-TOF	MM-ML	No	RecF	85 %	SB-EUPT	4	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
33		Yes	> 2 y	0.484	-0.5	0.05	10	ambient	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	iodoethane			SB-EUPT		$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
46		Yes	> 2 y	0.496	-0.5	0.01	25	ambient	No	No	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	No	None	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
20		Yes	> 2 y	0.5	-0.4	0.05	1	ambient	8 ml	No	mechanical shaking, 15 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No		GC-MSD		No	StAdd-SP	70 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
28		Yes	> 2 y	0.498	-0.4	0.05	50		50 ml	after H_2O, 5 min	45 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	MM-ML	No	None	76 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
98	x	Yes	> 2 y	0.509	-0.4	0.01	10	deep frozen	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-MSD	MM-ML	No		75 %	SB-EUPT	> 5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
104		No	< 1 y	0.508	-0.4	0.04	50	deep frozen	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	96 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
107		Yes	> 2 y	0.51	-0.4	0.05	2	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part., Filtration	GC-(P) FPD, Sulfur Filter	MM-ML	No	None	85 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
89		Yes	< 1 y	0.52	-0.3	0.05	10	ambient	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	MM-SL	No	PrCal	75 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
7		Yes	> 2 y	0.532	-0.2	0.05	50	ambient	No	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	No	None	70.2 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
49	x	No	> 2 y	0.53	-0.2	0.05	25	cold	No		mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane, toluene	HCl		No	Liq-liq part.	GC-MSD	PS-ML	No	None	62 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, no H_2O adittion
82		Yes	> 2 y	0.526	-0.2	0.1	50	ambient	No		30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	gaswashing	Spectrophotometer	PS-ML	No	None			4	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
11		Yes	> 2 y	0.547	-0.1	0.05	20	deep frozen				$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No	None	97 % (CS_2 = 0.253 mg/kg from Thiram-Solution)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
40		Yes	> 2 y	0.541	-0.1	0.05	20	ambient	Yes	after H_2O, 10 min	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Destillation	Spectrophotometer	PS-ML	No	None	92.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type); result not blank corrected
47	x	Yes	> 2 y	0.544	-0.1	0.05	50	ambient	No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	81.2 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
88		Yes	> 2 y	0.547	-0.1	0.02	50		No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No		87 % (thiram)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
102		Yes	> 2 y	0.55	-0.1	0.5	10		No	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	StAdd-SP	90 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
103		Yes	> 2 y	0.55	-0.1	0.3	100	ambient	Yes	after H_2O, 10 min		$SnCl_2$/HCl	H_2O/HCl	HCl				Spectrophotometer, UV	PS-ML	No	None	92 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
29	x	Yes	> 2 y	0.558	0.0	0.05	25	slightly frozen	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 25 ml isooctane	HCl		No	Dessication with Na_2SO_4	GC-(μ) ECD, Agilent	MM-ML	No	None	93 % (0.6 mg/kg)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
87		Yes	> 2 y	0.563	0.0	0.02	50	just thawed	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer, 272 nm, 302 nm, 332 nm	PS-ML	No	None	90.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
35		Yes	> 2 y	0.568	0.1	0.05	50	ambient	45 mL	No	manual shaking, 60 min	$SnCl_2$/HCl, water bath, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	PS-ML	No	None	83 % (0.550 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
55		Yes	> 2 y	0.568	0.1	0.02	50	deep frozen	45 mL H_2O	after H_2O, 20 min	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Centrifugation	GC-MS/MS (QQQ)	PS-ML	No	None	78 % (0.04 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
1		No	> 2 y	0.59	0.2	0.003	1	deep frozen	4 ml	No		$SnCl_2$/HCl, 15 min	H_2O/HCl	HCl		No		GC-MSD, Headspace	StAdd-SP	DCM	StAdd-SP	79 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
70	x	Yes	> 2 y	0.589	0.2	0.2	25	ambient	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl			No	GC-(P) FPD	MM-ML	No	None	80 %	SB-other	5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
99		Yes	> 2 y	0.59	0.2	0.3	75	ambient	No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	No	Spectrophotometer	PS-ML	No	None	70 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
73		Yes	> 2 y	0.599	0.3	0.05	10	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-(P) FPD	PS-ML	thiophene	None	95.6 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
81	x	Yes	> 2 y	0.616	0.4	0.05	50	cold	No		ultrasonic bath, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 25 ml 2.2,4-trimethyl pentane	HCl		No		GC-MSD	MM-ML	No	None	80 %	SB-other	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
86		Yes	> 2 y	0.617	0.4	0.05	20	slightly frozen	No	No	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	78 %	QC	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
2		Yes	> 2 y	0.635	0.5	0.05	80	deep frozen	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer		No					$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
68		Yes	> 2 y	0.63	0.5	0.05	200		200 mL			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No		84.4 %	SB-EUPT	2	PN-EN 12396-1: SnCl2/HCL-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
106		Yes	> 2 y	0.63	0.5	0.05	25	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None	105 % (matrix EUPT SRM7)	SB-other	1	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
19		Yes	> 2 y	0.65	0.6	0.05	25	ambient	No	No	30 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	NAOH+ H_2SO_4	Spectrophotometer, 272+302+332nm	PS-ML	No	None	100 % (0.050 + 0.50 mg/kg Thiram)	SB-other	2	$SnCl_2$/HCl-cleavage, KOH/MeOH, spectroph. analysis (Xanthogenate mth.) (EN 12396-3 type)
25		Yes	> 2 y	0.648	0.6	0.05	10	deep frozen	10 ml	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	83 % (0.411 mg/kg Thiram)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, headspace sampling of non-polar solvent
113		Yes	> 2 y	0.65	0.6	0.05	25	cold				$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	MM-ML	No	None	85 % (0.05. Thirum)	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
45		Yes	> 2 y	0.661	0.7	0.02	20	cold	No	No	60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 100 ml	HCl		No	No	GC-(P) FPD	StAdd-SP	No	None	104 % (0.3 mg/kg spiking level)	SB-EUPT	2	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
72		Yes	> 2 y	0.653	0.7	0.05	50	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		Yes	Pb(CH3COO)2	Spectrophotometer	PS-ML	No	None	81 %	SB-EUPT	3	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
115		Yes	None	0.66	0.7	0.025	5	deep frozen	No	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Centrifugation, Liq-liq part	GC-MS	PS-ML	generic IS	None	100 %		> 5	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
91	x	Yes	> 2 y	0.665	0.8	0.05	10	slightly frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-(P) FPD (following derivatization)	PS-ML	thiophene	None	68.8 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
34		Yes	> 2 y	0.688	0.9	0.5	50	deep frozen			manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl, 200 mL	1× (HCl)		Yes	No	Spectrophotometer, UV 435 nm	MM-ML	No	None		SB-EUPT	1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
3		Yes	> 2 y	0.698	1.0	0.02	2	cold	2 ml	after H_2O, min	mechanical shaking, 45 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-Ion Trap	MM-ML	No	None	100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
22		Yes	> 2 y	0.704	1.0	0.05	10	ambient	hydrolysis sln	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, 2 h, 70 °C	H_2O/HCl	1× (pH 1)		No		GC-(μ) ECD	MM-ML	No	None	99 % (CS_2, 0.5 mg/kg)	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
38		Yes	> 2 y	0.696	1.0	0.05	25	ambient	25 ml	No	manual shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(μ) ECD	MM-ML	No	StAdd-SP	87.4 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
61		Yes	> 2 y	0.7	1.0	0.01	3	cold	No		> 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl			No	GC-(μ) ECD	MM-ML	No	PrCal	117 %	QC		$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
51	x	Yes	> 2 y	0.706	1.1	0.05	30	slightly frozen	No		mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No		GC-MSD	PS-ML	No		86 %	SB-EUPT	4	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Dithiocarbamates (Assigned value = 0.559 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis / Cleavage step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
118		Yes	> 2 y	0.721	1.2	0.01	1	ambient	No	No		$SnCl_2$/HCl	H_2O/HCl	HCl				GC-MSD, HeadSpace sampling with GC/MS		No	None			4	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
9		Yes	> 2 y	0.774	1.5	0.05	25	cold	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl				GC-MSD	PS-ML	No	None	85 % (not corrected)	SB-EUPT	3	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
84	x	Yes	> 2 y	0.762	1.5	0.05	25	slightly frozen	No	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	PS-ML	No	None	85 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
78		Yes	> 2 y	0.81	1.8	0.01	2	ambient	No	No	manual shaking, 1 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MS/MS (QQQ)	PS-ML	generic IS	None	94 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
16		Yes	> 2 y	0.832	2.0	0.01	50	cold			manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)		No	Liq-liq part.; Iso-octane	GC-MSD, m/z: 76 and 78	PS-ML	No	None	82 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
114		No		0.84	2.0	0.05														other compound	None	83 %	SB-EUPT	1	no data
32		No	> 2 y	0.86	2.2	0.2	50	just thawed	200 mL	No		$SnCl_2$/HCl	H_2O/HCl	HCl		Yes		Spectrophotometer	PS-ML	No	None			1	$SnCl_2$/HCl-cleavage, Cu(II) acetate & DEA spectroph. analysis (EN 12396-1 /DFG S15-type)
43		Yes	> 2 y	0.87	2.2	0.02	1	ambient	Yes	No	mechanical shaking, 60 min	$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC-Ion Trap	MM-ML	No	PrCal				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
21		Yes	> 2 y	0.883	2.3	0.01	25	cold	Yes	No	mechanical shaking	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MSD	PS-ML	No		121 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
48		Yes	> 2 y	0.881	2.3	0.1	25	ambient	see hydrolysis	No	manual shaking, 5 min	HCl/$SnCl_2$	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)		No	Centrifugation	GC-MSD	PS-ML	No	None	93 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, GC-MS
116		Yes	> 2 y	0.88	2.3	0.5	6	ambient	No	No	60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	PS-ML	No	None	80 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
67	x	Yes	> 2 y	0.894	2.4	0.025	5	ambient	No	No	mechanical shaking, > 60 min	$SnCl_2$/HCl, water bath, 2 h, 80 °C	H_2O/$SnCl_2$/HCl-isooctane	HCl		No		GC-Ion Trap	PS-ML	No		100 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
76		Yes	1 - 2 y	0.89	2.4	0.05	25	ambient	Yes	No		HCl/$SnCl_2$	H_2O/$SnCl_2$/HCl-isooctane	1× (HCl)				GC-(μ) ECD	PS-ML	chloroform	None	76 %			$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2, Analysis of Dithiocarbamate Residues in Foods of Plant Origin Involving Cleavage into Carbon Disulfide, Partitioning into Isooctane
74	x	Yes	> 2 y	0.91	2.5	0.05	50	cold	Yes	No	manual shaking, 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-MSD	PS-ML	$^{13}CS_2$	None				$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
53		No	> 2 y	0.92	2.6	0.05	2	deep frozen	No			$SnCl_2$/HCl	H_2O/HCl	HCl		No	No	GC/ECD	MM-ML	No	PrCal				$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
4	x	No	None	0.95	2.8	0.05	50	slightly frozen	45 mL	No	mechanical shaking, > 60 min	HCl/$SnCl_2$, 2 h	H_2O/HCl	HCl		No	No	GC-MSD	PS-ML	No	None	55 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, headspace sampling, GC-Analysis of CS_2
63		Yes	> 2 y	1.03	3.4	0.1	2	ambient	No	No	manual shaking, 10 min	HCl/$SnCl_2$,	H_2O/HCl	HCl			No	GC-FID		other compound	PrCal	85 %	QC		$SnCl_2$/HCl-cleavage, headspace SPME, GC-Analysis of CS_2 (EN 12396-2 type)
64	x	Yes	> 2 y	1.03	3.4	0.05	25	deep frozen	No	No		$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	Liq-liq part.	GC-MSD	MM-ML	No	None	110 %	SB-EUPT	1	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
59	x	Yes	> 2 y	1.064	3.6	0.05	25	cold	No		mechanical shaking, > 60 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(μ) ECD	PS-ML	No	None	109 %	SB-EUPT	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2
85		No	> 2 y	1.25	4.9	0.05	5	deep frozen	No	No	manual shaking, 5 min	$SnCl_2$/HCl	H_2O/$SnCl_2$/HCl-isooctane	HCl		No	No	GC-(P) FPD	MM-ML	No		97 % (0.05 mg/kg)	QC	2	$SnCl_2$/HCl-cleavage, liq.-liq.-part. w. non-polar solvent, GC-Analysis of CS_2

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
118		No	None	0.0032	-3.9		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, negative	MM-SL	glyphosate-^{13}C	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
115		Yes			-3.5 (FN)	0.5	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML						QuEChERS-Citrate buffered (EN 15662), modified method
8		No	> 2 y	0.025	-3.4	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		FA				No	LC-MS/MS (QQQ)		ILIS	other	71 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
31		Yes	< 1 y	0.047	-2.8	0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	perclorarte ^{18}O	PrCal	100 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
77		No	> 2 y	0.059	-2.5	0.01												no data		No	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.0677	-2.3		5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	87 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.07	-2.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
75		No	None	0.082	-2.0	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4	StAdd-SP	115 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
78		Yes	> 2 y	0.083	-1.9	0.01	5	ambient	No	No	ultra turrax, 1 min	No	EtOAc	1×		Yes	Dessication with $MgSO_4$	GC-MS/MS (QQQ) (following cleavage)	MM-ML	generic IS	None	115 %	SB-EUPT	1	Method involv. ethylene-release (§ 64 LFGB 00.00-47-type)
113		Yes	> 2 y	0.083	-1.9	0.05	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	76 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
117		Yes	> 2 y	0.114	-1.2	0.01	2	just thawed	Yes	after H_2O, min	manual shaking		Isooctane					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77 %	SB-EUPT	1	O-tins: QuEChERS-based mth by EURL-SRM
67	x	No	None	0.1238	-0.9	0.05	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		102 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
103		Yes	1 - 2 y	0.124	-0.9	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 10 min		MeOH + 1 % FA	No			No	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	72 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.13	-0.8	0.02	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.128	-0.8	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		No	< 1 y	0.133	-0.7	0.06	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	85.9 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:20
94	x	No	None	0.133	-0.7	0.1	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	78.2 % (0.1 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
16		Yes	> 2 y	0.139	-0.6	0.05	5	cold	9.3 mL	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	108.4 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	> 2 y	0.139	-0.6	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	PrCal	117 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
34		Yes	> 2 y	0.137	-0.6	0.05	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	PrCal	111 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	Yes	> 2 y	0.137	-0.6	0.02	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	None	95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.141	-0.5	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ), 143/107	PS-ML	ethephon-D_4	PrCal	80 % (96 % D_4-Ethephon recovery)	SB-EUPT	1	extraction with MeOH, LC-MS/MS detection
58		No	1 - 2 y	0.146	-0.4	0.02	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
72		No	1 - 2 y	0.146	-0.4	0.02	10	ambient	No	after H_2O, 5 min	mechanical shaking, 5 min	No	Isooctane	No		derivated by diazomethane	No	GC-(P) FPD (following derivatization)	MM-ML	No	None	77 %	SB-EUPT	3	other (derivated by diazomethane)
2		Yes	> 2 y	0.151	-0.3	0.02	10	deep frozen	No	No	mechanical shaking, 60 min	No	MeOH / H_2O / FA	No		No	Disp.-SPE; carbon	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	85 % (0.2 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
3		Yes	> 2 y	0.148	-0.3	0.05	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		No	None	0.15	-0.3	0.05	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
45		Yes	> 2 y	0.148	-0.3	0.02	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	107 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.15	-0.3	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	101 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	0.15	-0.3	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	80 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial
27		Yes	> 2 y	0.153	-0.2	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	100 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
47	x	No	None	0.155	-0.2	0.1	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Freezing out	LC-MS/MS (QQQ)	MM-SL	No	None	72.1 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.152	-0.2	0.05	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	100 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	Yes	> 2 y	0.157	-0.1	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
21		Yes	> 2 y	0.159	-0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
23	x	Yes	1 - 2 y	0.158	-0.1	0.02	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	93 %	SB-EUPT	3	other (EN 15055, 2006-08)
85		No	1 - 2 y	0.156	-0.1	0.02	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		119 % (0.02 mg/kg)	QC	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.162	0.0	0.02	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	MM-ML	ethephon-D_4		99 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
14		No	None	0.162	0.0	0.02	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	87 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
28		No	1 - 2 y	0.164	0.1	0.02	5		10 ml	after H_2O, 5 min	mechanical shaking, 5 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml					LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	73 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
12	x	Yes	> 2 y	0.169	0.2	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	StAdd-EA	105.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.168	0.2	0.01	5	ambient	10 ml	No	mechanical shaking, 60 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	None	113 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.178	0.4	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		ethephon-D_4	StAdd-SP	88 %	SB-EUPT	2	Accreditation in request
10		Yes	> 2 y	0.185	0.6	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	None	104 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
51	x	Yes	> 2 y	0.185	0.6	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		106 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
91	x	No	> 2 y	0.187	0.6	0.02	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), 1 daughter ion	MM-ML	No	None	78.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no ISTD was used
15		Yes	> 2 y	0.193	0.8	0.02		ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	90 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), weight 2.5 g of sample
22		No	None	0.193	0.8	0.05	10	ambient	140 ml	No	mechanical shaking, > 60 min	No		1× (pH 14)		Yes		GC-FID (following derivatizsation)	MM-ML	Aceton	None	102 % (0.2 mg/kg)	SB-EUPT	1	Method involv. ethylene-release (§ 64 LFGB 00.00-47-type); GC-Headspace
64	x	No	> 2 y	0.196	0.8	0.04	5	deep frozen	10 ml	after H_2O, 10 min		No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	299.8 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
89		Yes	< 1 y	0.194	0.8	0.01	5	ambient	25 ml	after H_2O, 5 min	mechanical shaking, 10 min		DCM	1× (FA)				LC-MS/MS (QQQ)	MM-ML	ioxynil	PrCal	60 %	SB-EUPT	3	other (H_2O / DCM)
82		Yes	> 2 y	0.2	0.9	0.05	5	ambient	10 ml	No	manual shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.21	1.2	0.02	10	ambient	No	No	mechanical shaking, 10 min	No	ACN, MeOH 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4					in house
84	x	Yes	> 2 y	0.212	1.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (as for QuPPe)		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	106 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.215	1.3	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	ethephon-D_4	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.215	1.3	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	71 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
29	x	Yes	< 1 y	0.218	1.4	0.05	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), Agilent	MM-ML	ethephon-D_4	PrCal	102 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.224	1.5	0.02	5	ambient	5 ml	No	manual shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), DCM	No		No	No	LC-MS	MM-ML	No	StAdd-SP	80.6 %	SB-EUPT	1	lab method
57		No	> 2 y	0.221	1.5	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	84 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Ethephon (Assigned value = 0.162 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
100		Yes	> 2 y	0.23061	1.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	56.99 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
35		Yes	> 2 y	0.24	1.9	0.02	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None	133 % (0.23 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	Yes	> 2 y	0.241	2.0	0.05	5	cold	Yes	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	ethephon-D_4	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
55		Yes	< 1 y	0.27	2.7	0.02	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	ethephon-D_4	PrCal	107 % (0.04 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
116		Yes	1 - 2 y	0.28	2.9	0.02	2	ambient	No	No	mechanical shaking, 5 min	No	ACN, H_2O	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	102 %	SB-EUPT	1	other (extraction with ACN:H_2O)
44	x	Yes	> 2 y	0.351	4.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
26	x	No	1 - 2 y	0.388	5.6		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	104.2 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
36		Yes	> 2 y	0.411	6.2	0.02	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_4 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
13		Yes			-3.6 (FN)	0.01												no data							Method involv. deriv. w. FMOC
26	x	No			-3.6 (FN)	0.5	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		FMOC	No	LC-MS/MS (QQQ)	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
115		Yes			-3.6 (FN)	0.01	1	deep frozen	10 ml	No	mechanical shaking, 20 min	No	DCM, H_2O	2× (sodium borate)		FMOC over-night	Centrifugation, SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML						Method involv. deriv. w. FMOC
118		No			-3.6 (FN)		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, negative	MM-SL						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		Yes	1 - 2 y	0.229	-2.4	0.2	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
45		No	1 - 2 y	0.241	-2.3	0.05	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	93 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
91	x	No	> 2 y	0.242	-2.3	0.05	20	slightly frozen	No		ultra turrax, 5 min	No	H_2O, 100 ml H_2O	No		with triflouoro-aceticacid anhydride and fluorated buthanole	Liq-liq part., SPE-column (ion exchange), LLE with DCM	LC-MS/MS (QQQ), 4 daughter ions	PS-ML	No	None	66.5 %	SB-EUPT	1	other (with derivatization), 1) extraction with H_2O, 2) clean-up, 3) derivatization
8		No	> 2 y	0.262	-2.2	0.05	1	ambient	9 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	84 %	SB-EUPT	2	Method involv. deriv. w. FMOC
72		No	1 - 2 y	0.264	-2.1	0.05	25	ambient	No	No	mechanical shaking, 20 min	No	H_2O	No		derivated by TMOAc	No	GC-(P) FPD (follow-ing derivatization)	PS-ML	No	RecF	53 %	SB-EUPT	3	other (derivated by TMOAc)
61		Yes	> 2 y	0.3	-1.9	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	92 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
32		No	> 2 y	0.34	-1.6	0.05	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	0.391	-1.2	0.1	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	55 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial
48		Yes	> 2 y	0.406	-1.1	0.03	20	ambient	see pH adjust-ment	No	mechanical shaking, 30 min	No	DCM	more than 2× (HCl/HCl/ buffer)		OPA	Centrifugation, SPE-column (ion exchange)	LC-FLD (Fluorescence)	PS-ML	No	None	86 %	SB-EUPT	2	Method involv. post-colum deriv. w. OPA (DFG-405 type)
11		Yes	> 2 y	0.421	-1.0	0.05	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$		120 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
78		Yes	> 2 y	0.42	-1.0	0.05	3	ambient		No	ultra turrax, 1 min	No	Isooctane, H_2O	No		Yes	Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS	None	90 %	SB-EUPT	1	Method involv. deriv. w. FMOC
89		No	< 1 y	0.442	-0.9	0.05	5	ambient	25 ml	after H_2O, 5 min	mechanical shaking, 10 min		DCM	1× (FA)				LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	88 %	SB-EUPT	3	other (H_2O / DCM)
21		Yes	> 2 y	0.453	-0.8	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	93 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
54	x	Yes	> 2 y	0.469	-0.7	0.02	1	ambient	No	No	ultra turrax, 1 min	No	H_2O, Isooctane, DCM	No		FMOC	Centrifugation, Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	other	92 %	SB-EUPT	1	Method involv. deriv. w. FMOC, Goscinny et al, Food Analytical Methods 2012;5;1177-1185

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
82		Yes	> 2 y	0.462	-0.7	0.1	5	ambient	10 ml	No	manual shaking, 5 min	No	Isooctane	2× (ammonia, FA)		deriv. w. isobutyl-chloroformate	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	Method involv. deriv. w. FMOC
94	x	No	None	0.472	-0.7	0.1	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	44.6 % (0.1 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.484	-0.6	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.495	-0.5	0.05	3	ambient	25 ml	No	ultrasonic bath, 10 min	No	H_2O	No		No	No	LC-MS	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	lab method
3		Yes	1 - 2 y	0.506	-0.4	0.04	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	108 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
75		Yes	> 2 y	0.517	-0.4	0.05	2	ambient	see solvent details	after H_2O and organic solvent, 30 min	ultrasonic bath, 30 min	No	EtOAc, H_2O/MeOH/Borat-buffer 6:3:1	1× (after SPE-elution to pH 9 with NH_3)		with fluorenyl-methyl)-chlorformiat	SPE-column; Oasis MAX, 30 Åµm; elution with ACN/HCl	LC-MS/MS (QQQ)	StAdd-SP	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-SP	106 %	SB-EUPT	2	Method involv. deriv. w. FMOC
4	x	Yes	> 2 y	0.527	-0.3	0.05	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
51	x	Yes	> 2 y	0.526	-0.3	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	ILIS	StAdd-EA	87 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
55		Yes	> 2 y	0.53	-0.3	0.01	3	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	Isooctane, DCM	1× (pH 9)		FMOC	SPE-column (C18)	LC-MS/MS (QQQ)	MM-ML	glyphosate $^{13}C^{15}N$	PrCal	103 % (0.02 mg/kg)	SB-EUPT	1	Method involv. deriv. w. FMOC
116		Yes	> 2 y	0.52	-0.3	0.1	2	ambient	No	No	ultra turrax, 1 min	No	H_2O	No		FMOC	No	LC-MS/MS (QQQ)	PS-SL	No	None	73 %	SB-EUPT	2	Method involv. deriv. w. FMOC
95		No	< 1 y	0.536	-0.2		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.55	-0.1	0.05	1	ambient	10 ml	No	mechanical shaking, 60 min	Yes	H_2O	No		FMOC-Cl	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	Method involv. deriv. w. FMOC
20		Yes	1 - 2 y	0.55	-0.1	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		glyphosate 2	StAdd-SP	110 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
74	x	Yes	> 2 y	0.55	-0.1	0.01	3	cold	Yes	No	ultrasonic bath, 20 min	No	H_2O	No		FMOC	Liq-liq part.	LC-MS/MS (QQQ)	MM-ML	$1,2\text{-}^{13}C_2\ ^{15}N$-glyphosate	None				Method involv. deriv. w. FMOC
6	x	Yes	> 2 y	0.568	0.0	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.564	0.0	0.01		ambient	No		mechanical shaking, 30 min	No	0.1 mol/l HCl	1×		after cleanup derivatisation with FMOC	Centrifugation; Cleanup 2 with Dichlormethan	LC-MS/MS (QQQ), 390/168	PS-SL	$^{13}C,^{15}N$ glyphosate	PrCal	88 % (88 % $^{13}C.\ ^{15}N$ Glyphosat recovery)	SB-EUPT	1	Method involv. deriv. w. FMOC, C18 clean up
12	x	Yes	> 2 y	0.582	0.1	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-EA	103.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
16		Yes	1 - 2 y	0.576	0.1	0.05	3	ambient	25 + 25 mL	No	ultrasonic bath, 10 min	No	H_2O, DCM: 2-propanol	2× (alkaline/acidic)		FMOC	DCM:2-propanol	LC-MS/MS (QQQ)	MM-ML	glyphosate-^{13}C	PrCal	107.2 %	SB-EUPT	2	Method involv. deriv. w. FMOC
57		No	1 - 2 y	0.584	0.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	66 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
114		Yes		0.58	0.1	0.05														ILIS	StAdd-SP	120 %	SB-EUPT	1	no data

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
35		Yes	> 2 y	0.598	0.2	0.02	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	glyphosate-^{15}N-^{13}C	None	110 % (0.559 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.593	0.2	0.1	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
44	x	Yes	> 2 y	0.592	0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	1 - 2 y	0.592	0.2	0.05	5	ambient	10 ml	No	mechanical shaking, 60 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	106 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
52		Yes	1 - 2 y	0.602	0.2	0.05	5	just thawed	10 ml	after H_2O, 10 min	mechanical shaking, 30 min	No		No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$		101.2 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), 0.5 % FA in Eluent A and B, freezing out of MeOH extract
81	x	Yes	> 2 y	0.603	0.2	0.05	5	cold	9.5 ml		mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	glyphosate (^{13}C, ^{15}N)	None	103 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
111		Yes	> 2 y	0.597	0.2	0.05	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	96 % (2x MRRL)	SB-EUPT	1	other
2		Yes	1 - 2 y	0.616	0.3	0.05	3	deep frozen	9.75 mL	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	PS-ML	glyphosate $^{13}C^{15}N$	None	101 % (0.5 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Cf. extraction, clean up and chromatography
22		Yes	> 2 y	0.607	0.3	0.05	10	ambient	No	No	ultrasonic bath, 20 min	No	DCM, part. with 20 ml HCl, 10 ml DCM	1× (neutralisation with NaOH to pH 6-8)		FMOC	Centrifugation; ion-exchange	LC-FLD (Fluorescence)	MM-SL	No	PrCal	rec. spiking level = calibr. level, (derivatis. step is included, rec. figure not posible)	SB-EUPT	3	Method involv. deriv. w. FMOC, acid extraction,neutralisation,ion-exchange,LC-Fluorescense-Detection after derivatisation with FMOC
65	x	No	None	0.612	0.3	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	88 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
18		Yes	> 2 y	0.62	0.4	0.05	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		90 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	> 2 y	0.623	0.4	0.02	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	PrCal	103 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
29	x	Yes	1 - 2 y	0.619	0.4	0.01	5	slightly frozen	No		manual shaking, 2 min		H_2O, DCM, H_2O+0.1 %FA	1× (pH 6)		FMOC	SPE-column; Oasis HLB	LC-MS/MS (QQQ), Agilent	MM-ML	glyphosate $^{13}C^{15}N$	PrCal	101 %	SB-EUPT	4	Method involv. deriv. w. FMOC
33		Yes	> 2 y	0.62	0.4	0.05	3	ambient	No	No	mechanical shaking, 60 min	No	H_2O 0.1 FA, H_2O 1 % FA	No		adding FMOC and Borate buffer	SPE-column (C18)	LC-MS/MS (QQQ)	StAdd-SP	No					Method involv. deriv. w. FMOC
10		Yes	> 2 y	0.639	0.5	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	108 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.64	0.5	0.04		ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	96 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), weight 2.5 g of sample
28		Yes	> 2 y	0.651	0.6	0.05	5		10 ml	after H_2O, 5 min	mechanical shaking, 5 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml					LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	None	72 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
58		No	1 - 2 y	0.653	0.6	0.05	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

COMPULSORY ANALYTES | Glyphosate

Glyphosate (Assigned value = 0.568 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
84	x	Yes	> 2 y	0.652	0.6	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (as for QuPPe)		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	98 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
27		Yes	> 2 y	0.67	0.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	109 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
106		No	< 1 y	0.67	0.7	0.5	1	deep frozen										LC-Orbitrap	MM-ML	ILIS	None	99 % (matrix EUPT SRM7)	SB-other	1	Method involv. deriv. w. FMOC
36		Yes	> 2 y	0.676	0.8	0.05	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
1		No	1 - 2 y	0.697	0.9	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	1× (Borate buffer)		FMOC-Cl	Liq-liq part.; ethylacetatee/ CyH 1/1	LC-MS/MS (QQQ)	MM-ML	ILIS	StAdd-SP	98 %	SB-EUPT	1	Method involv. deriv. w. FMOC
83		Yes	> 2 y	0.707	1.0	0.04	25	ambient			mechanical shaking, 30 min	No	H_2O/HCl, DCM, 0.1M HCl	1× (pH between 1.6 and 2.4)			SPE-column (ion exchange), SPE-column (ion exchange), SPE 1 = Chelex 100; SPE2 = AG 1-X8	LC-FLD (Fluorescence), postcolumn derivatisation (OPA)	PS-ML	No	RecF	50 %	SB-EUPT	3	Method involv. post-colum deriv. w. OPA (DFG-405 type)
64	x	No	> 2 y	0.746	1.3	0.08	5	deep frozen	10 ml	after H_2O, 10 min		No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	107 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.772	1.4	0.5	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	ILIS	None	97 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.775	1.5	0.02	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	glyphosate $1,2\text{-}^{13}C_2\ ^{15}N$	StAdd-EA	119 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
23	x	Yes	1 - 2 y	0.788	1.6	0.05	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	96 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.84408	1.9	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	72.8 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	2.494	13.6	0.05	2	ambient										LC-MS/MS (QQQ)	StAdd-SP	No	StAdd-SP	100 %			Method involv. deriv. w. FMOC; detected in blank at high concentration.
34		Yes	> 2 y	2.85	16.1	0.04	5	deep frozen	No		mechanical shaking, 15 min	No	H_2O, 20 mL	No		FMOC	Liq-liq part.; EtOAc	LC-MS/MS (QQQ), ESI neg	MM-ML	No	PrCal	73 %	SB-EUPT	1	Method involv. deriv. w. FMOC

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
1		No			-3.5 (FN)	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML						QuEChERS-Citrate buffered (EN 15662)
34		No	> 2 y	0.016	-3.2	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Centrifugation	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	76 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
115		Yes	None	0.02	-3.0	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
24	x	No	> 2 y	0.0294	-2.6	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
114		No		0.029	-2.6	0.02														other compound	StAdd-SP	104 %	SB-EUPT	1	no data
64	x	Yes	> 2 y	0.038	-2.1		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	40.4 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
78		Yes	> 2 y	0.047	-1.7	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	65 %	SB-EUPT	2	in house method
97	x	No	> 2 y	0.053	-1.4	0.02	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/$MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	nicarbazin	None	74 % (0.02 mg/kg)	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM, EURL SRM
89		Yes	< 1 y	0.057	-1.2	0.005	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	84 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
18		Yes	> 2 y	0.058	-1.1	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.061	-1.0	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column (ion exchange)	LC-MS/MS (QQQ)	PS-ML	MCPP-D_3	PrCal	98 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
16		Yes	> 2 y	0.0612	-1.0	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	63.5 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
58		Yes	> 2 y	0.061	-1.0	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
65	x	Yes	> 2 y	0.0656	-0.8	0.01	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	69 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
75		Yes	> 2 y	0.064	-0.8	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	MCPA D_6	StAdd-EA	63 %	QC	> 5	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.066	-0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
118		No	None	0.0667	-0.7		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
28		No	> 2 y	0.068	-0.6	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	106 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
32		Yes	> 2 y	0.068	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
35		Yes	> 2 y	0.0688	-0.6	0.01	5	ambient	10 ml	No	mechanical shaking, 30 min	No	EtOAc with 1 % HOAc	1× (1 % HOAc)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	80 % (0.101 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
59	x	Yes	> 2 y	0.071	-0.5	0.05	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min	NaOH	ACN	1× (H_2SO_4)		Yes	C18/$MgSO_4$	GC-MSD (following derivatization)	MM-ML	No	None	75 %	SB-EUPT	2	other (with derivatization)
85		No	> 2 y	0.07	-0.5	0.01	5	cold	10 ml	after H_2O and organic solvent, 30 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		80 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
40		Yes	> 2 y	0.074	-0.4	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifugation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	80 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
54	x	Yes	> 2 y	0.073	-0.4	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	90 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 with extract conc
57		Yes	> 2 y	0.072	-0.4	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	129 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
76		Yes	1 - 2 y	0.073	-0.4	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No				LC-Orbitrap	PS-ML	2.4,6-trichloro-phenol	None	82 %			A-QuEChERS (with 1 % FA)
30		Yes	> 2 y	0.075	-0.3	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabutyl-ammoniumhydroxide/iodomethane		GC-MSD (following derivatization), m/z:214/216/141	PS-SL	No	None	88 %	SB-EUPT	1	alkaline hydrolysis extraction, GPC, acid/base distribution, methylation, GC-MSD detection
31		Yes	< 1 y	0.075	-0.3	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	46 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
56		Yes	> 2 y	0.0741	-0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	91 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.0741	-0.3	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	80 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
91	x	Yes	> 2 y	0.0752	-0.3	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.7 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
70	x	No	> 2 y	0.0767	-0.2	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	60 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.078	-0.2	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	None			4	QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.078	-0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
26	x	No	> 2 y	0.08	-0.1		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	70.8 %		3	QuEChERS-Citrate buffered (EN 15662)
37	x	Yes	> 2 y	0.079	-0.1	0.05	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	102 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA cleaning
43		Yes	> 2 y	0.08	-0.1	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA				QuEChERS-Citrate buffered (EN 15662)
45		Yes	> 2 y	0.0795	-0.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	99 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
72		Yes	> 2 y	0.0798	-0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
84	x	No	None	0.079	-0.1	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
106		Yes	> 2 y	0.079	-0.1	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	93 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
107		Yes	> 2 y	0.0792	-0.1	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.081	0.0	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)		ILIS	other	83 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM
11		Yes	> 2 y	0.082	0.0	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		105 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
15		Yes	> 2 y	0.0804	0.0	0.004	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	93 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
20		Yes	> 2 y	0.0815	0.0	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	76 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
21		Yes	> 2 y	0.082	0.0	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	98 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.081	0.0	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	95 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
38		Yes	> 2 y	0.081	0.0	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	87.8 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
53		No	> 2 y	0.081	0.0	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	104 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
90		Yes	> 2 y	0.0805	0.0	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	92 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
47	x	No	None	0.084	0.1	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	80.6 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
80		Yes	> 2 y	0.083	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
33		Yes	> 2 y	0.085	0.2	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
87		Yes	> 2 y	0.086	0.2	0.01	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	70.6 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
25		Yes	> 2 y	0.087	0.3	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	85 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
98	x	Yes	1 - 2 y	0.087	0.3	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		98 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)

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- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
103		Yes	1 - 2 y	0.087	0.3	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	68 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
3		Yes	> 2 y	0.09	0.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	116 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
19		Yes	1 - 2 y	0.09	0.4	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
22		Yes	> 2 y	0.0891	0.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4), PSA/ $MgSO_4$ (pH > 8))		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	98 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
116		No	1 - 2 y	0.09	0.4	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
12	x	Yes	1 - 2 y	0.091	0.5	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	105.7 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.091	0.5	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	101 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
66		Yes	1 - 2 y	0.091	0.5	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	98 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
73		No	None	0.0912	0.5	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	94.9 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	2.5-dichloroben-zoic acid	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
10		Yes	> 2 y	0.0957	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	95 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
46		Yes	1 - 2 y	0.095	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	93 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
60	x	No	< 1 y	0.0961	0.7	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	95 %	SB-EUPT	3	Modified QuEChERS
79		No	< 1 y	0.096	0.7	0.05	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	107 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
94	x	Yes	> 2 y	0.0963	0.7	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	103 % (0.01 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
36		Yes	> 2 y	0.0964	0.8	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	RecF	68 % (Avegrage from ongoing AQC samples)	QC	> 5	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

MCPA (Assigned value = 0.081 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
23	x	Yes	1 - 2 y	0.1	0.9	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	110 %	SB-EUPT	3	other
61		Yes	> 2 y	0.1	0.9	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	97 %	QC		QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	> 2 y	0.102	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
113		Yes	> 2 y	0.104	1.1	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	110 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.1076	1.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		89 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
55		Yes	> 2 y	0.11	1.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	PrCal	88 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
100		Yes	> 2 y	0.11445	1.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	133.61 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.115	1.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	106 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.122	2.0	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	45 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
4	x	No	None	0.125	2.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	$MCPA\ D_3$	StAdd-SP			1	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.135	2.7	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)

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deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

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Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
118		No	None	0.0154	-3.5		5	ambient	Yes	after H_2O, 10 min	15 min	No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-SL	chlormequat-D_4	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
85		No	< 1 y	0.026	-3.1	0.01	5	cold	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No		60.2 % (0.05 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
97	x	No	> 2 y	0.065	-1.7	0.05	10	ambient	until volume to 20 mL	No	mechanical shaking, 2 min	No	Isooctane, 40 mL MeOH	No		No	No	LC-MS/MS (QQQ), internal standard	MM-SL	mepiquat iodide D_3	None	67 % (0.05 mg/kg)	SB-EUPT	2	analysis of chlormequat and mepiquat residue in foods of plant origin EURLSRM Jan 2009
78		Yes	> 2 y	0.071	-1.5	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	69 %	SB-EUPT	1	in house method
19		Yes	1 - 2 y	0.076	-1.3	0.05	5	ambient	No	No	5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	73.3 % (0.050 and 0.50 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
59	x	No	< 1 y	0.087	-0.9	0.02	10	cold		after H_2O, 10 min	manual shaking, 2 min	No	Isooctane	No		No	Centrifugation	LC-Ion Trap	PS-ML	ILIS		84 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
90		Yes	> 2 y	0.0881	-0.9	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane, acidified with FA	1× (by FA within the solvent)		No	Filtration; 0.45 µm	LC-MS/MS (QQQ)	MM-ML	No	None	73 % (0.08 mg/kg)	SB-EUPT	1	§ 64 LFGB, L00.00-76
113		Yes	> 2 y	0.089	-0.9	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	74 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
18		Yes	> 2 y	0.09	-0.8	0.01	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
111		Yes	> 2 y	0.09	-0.8	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	chlormequat-D_4	None	89 % (2x MRRL)	SB-EUPT	1	other
8		No	> 2 y	0.093	-0.7	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	85 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
36		Yes	> 2 y	0.0946	-0.7	0.01	5	ambient	5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	PS-ML	D_3 labelled					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
98	x	Yes	1 - 2 y	0.095	-0.7	0.01	5	deep frozen	Yes	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Centrifugation	LC-MS/MS (QQQ)	MM-ML	No		70 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
11		Yes	> 2 y	0.1	-0.5	0.01	10	deep frozen	10 ml	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ)	MM-ML	D_3-mepiqua-tiodid		102 % (0.1 and 0.2 mg/kg)	SB-EUPT	2	other
27		Yes	> 2 y	0.1	-0.5	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	111 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
57		No	1 - 2 y	0.1	-0.5	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	72 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	Yes	> 2 y	0.1	-0.5	0.01	25	cold	Yes	after H_2O, 10 min	ultra turrax, 2 min	No	Isooctane	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	PS-ML	D_3-mepiquat	None				MeOH/H_2O extraction
89		Yes	< 1 y	0.1	-0.5	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min		Isooctane	1× (FA)				LC-MS/MS (QQQ)	MM-ML	TPP	PrCal	98 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
114		Yes		0.1	-0.5	0.05														other compound	StAdd-SP	86 %	SB-EUPT	1	no data
3		Yes	> 2 y	0.101	-0.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
14		No	None	0.101	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
40		Yes	> 2 y	0.102	-0.4	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
75		Yes	> 2 y	0.103	-0.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	mepiquat D_3	StAdd-EA		QC	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
12	x	Yes	> 2 y	0.106	-0.3	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	StAdd-EA	94.8 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	> 2 y	0.105	-0.3	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		chlormequat chloride D_4	StAdd-SP	80 %	SB-EUPT	2	MeOH extraction
29	x	Yes	> 2 y	0.105	-0.3	0.01	5	slightly frozen	No		mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×		No	Centrifugation, Filtration, nylon filter	LC-MS/MS (QQQ), Waters	MM-ML	No	None	102 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
47	x	Yes	> 2 y	0.106	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	Isooctane				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	75.3 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
84	x	Yes	> 2 y	0.106	-0.3	0.01	10	slightly frozen	Yes	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	PS-ML	ILIS	None	113 %	SB-EUPT	1	EN15055
107		Yes	> 2 y	0.104	-0.3	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	ILIS	None	75 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
38		Yes	> 2 y	0.107	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	Isooctane	No		No	No	LC-MS	MM-ML	No	StAdd-SP	82.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
43		Yes	> 2 y	0.109	-0.2	0.008	5	ambient	Yes	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	PS-ML	D_3-mepiquat					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), freezing out
45		Yes	> 2 y	0.109	-0.2	0.005	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	102 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	Yes	> 2 y	0.108	-0.2	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	103 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
1		No	> 2 y	0.112	-0.1	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	21 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
5		Yes	> 2 y	0.11	-0.1	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
34		Yes	> 2 y	0.11	-0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	> 2 y	0.11	-0.1	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	CCC	PrCal	104 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	> 2 y	0.11	-0.1	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	91 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
83		No	< 1 y	0.11	-0.1	0.01	10	ambient			mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), using 120 ml of extraction solution

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

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Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
91	x	No	> 2 y	0.11	-0.1	0.01	5	slightly frozen	8.5 ml	No	mechanical shaking, 5 min	No	1 % FA in ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	No	None	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), solvent = 1 % FA in ACN, no ISTD was used
106		Yes	> 2 y	0.11	-0.1	0.01	10	deep frozen	Yes		15 min	No						LC-MS/MS (QQQ)	MM-ML	ILIS	None	93 %	SB-EUPT	1	other (extraction with MeOH)
6	x	Yes	> 2 y	0.115	0.0	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
9		Yes	> 2 y	0.114	0.0	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				Filtration	LC-MS/MS (QQQ)	PS-ML	isotope D_3	PrCal	106 % (not corrected)	SB-EUPT	3	SIST EN 15055:2006
16		Yes	< 1 y	0.115	0.0	0.01	5	ambient	10 ml	No	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	mepiquat iodide D_3	None	114.2 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
21		Yes	> 2 y	0.114	0.0	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	93 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
35		Yes	> 2 y	0.114	0.0	0.005	10	ambient	20 ml	No	mechanical shaking, 15 min	No	Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	mepiquat iodide D_3	None	102 %	SB-EUPT	1	Startin J.R., Hird S.J., Sykes M.D., Taylor J.C. and Hill A.J. Determination of residues...
46		Yes	1 - 2 y	0.115	0.0	0.005	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	108 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		Yes	> 2 y	0.116	0.1	0.004	5	ambient	No	after H_2O and organic solvent, 10 min	mechanical shaking, 20 min	No		1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	ILIS	RecF	79 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), extraction mixture: 20 mL 80 % ACN in H_2O, 1 % FA
51	x	Yes	> 2 y	0.117	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	ILIS		113 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
64	x	No	> 2 y	0.116	0.1	0.02	5	deep frozen	10 ml	after H_2O, 10 min		No	H_2O/HCl, Isooctane	No		No	Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	104.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
23	x	Yes	1 - 2 y	0.12	0.2	0.01	5		10 ml	after H_2O, 30 min	1 min		Isooctane	No				LC-MS/MS (QQQ)	PS-ML	ILIS	StAdd-SP	93 %	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
26	x	No	> 2 y	0.12	0.2		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1×			No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	86 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
54	x	Yes	> 2 y	0.12	0.2	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	StAdd-SP	No	StAdd-SP	75 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66 without dilution
77		No		0.12	0.2	0.01												no data		No	StAdd-SP				no data
2		Yes	> 2 y	0.122	0.3	0.01	10	deep frozen	No	No	mechanical shaking, 60 min	No	H_2O / MeOH / HCl	No		No	Disp.-SPE; alumina	LC-MS/MS (QQQ)	PS-ML	mepiquat D_3	None	89 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
56		Yes	1 - 2 y	0.122	0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/ $MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	36 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
66		Yes	> 2 y	0.122	0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	105 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
4	x	Yes	> 2 y	0.124	0.4	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	mepiquat D_3	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
55		Yes	> 2 y	0.126	0.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	115 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
87		Yes	> 2 y	0.126	0.4	0.02	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Centrifugation	LC-MS/MS (QQQ), ESI pos.	MM-ML	mepiquat D_3	None	102.4 %	SB-EUPT	1	§ 64 LFGB, L00.00-76
58		No	> 2 y	0.127	0.5	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Mepiquat (Assigned value = 0.114 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
94	x	Yes	> 2 y	0.13	0.6	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	101.9 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
116		Yes	> 2 y	0.13	0.6	0.01	2	ambient	Yes	after H_2O and organic solvent, 5 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	87 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
30		Yes	> 2 y	0.135	0.8	0.005	5	ambient	19.5 mL	after H_2O and organic solvent, 5 min	ultra turrax, 2 min	No	Isooctane	No		No		LC-MS/MS (QQQ), 114/98	PS-ML	D_3-mepiquat		94 % (87 % D_3-Mepiqat recovery)	SB-EUPT	1	DIN EN 15055, 2006-08
81	x	Yes	> 2 y	0.137	0.8	0.01	10	cold	19 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	equat chloride D_4	None	115 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	> 2 y	0.136	0.8	0.01	5	ambient	10 ml	No	ultrasonic bath, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI+	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
10		Yes	> 2 y	0.139	0.9	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
32		No	> 2 y	0.14	0.9	0.01	5	just thawed	10 ml	after H_2O and organic solvent, 20 min	manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (0.1 % FA in MeOH)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	StAdd-EA	StAdd-EA				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
44	x	Yes	> 2 y	0.142	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	Isooctane			No	Freezing out	LC-MS/MS (QQQ)		ILIS	StAdd-SP				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
80		Yes	> 2 y	0.142	1.0	0.01	10	ambient	10 ml	after H_2O and organic solvent, 10 min	ultra turrax, 1 min	No	ACN	No		No	SPE-column; OASIS WCA	LC-MS/MS (QQQ), Waters Xevo	StAdd-SP	mepiquat D_3	StAdd-SP				other
25		Yes	> 2 y	0.146	1.1	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	PrCal	107 % (0.05 mg/kg)	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
65	x	No	None	0.154	1.4	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	None	73 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	> 2 y	0.157	1.5	0.01	5	ambient	5 ml	No	ultra turrax, 1 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	mepiquat D_3					in house method
31		Yes	< 1 y	0.16	1.6	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	106 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	1 - 2 y	0.173	2.1	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN	Yes (HOAc buffered)			Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	StAdd-SP	50 %	SB-EUPT		QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
76		Yes	> 2 y	0.172	2.1	0.01	10	ambient	Yes	after H_2O and organic solvent, 1 min	1 min	No	MeOH	1×				LC-Orbitrap	PS-ML	atrazine	None	117 %			QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Analysis of Dithi-ocarbamate Residues in Foods of Plant Origin Involving Cleavage into Carbon Disulfide, Partitioning into Isooctane
67	x	Yes	> 2 y	0.1762	2.2	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		96 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
95		No	< 1 y	0.214	3.5		5	ambient	10 ml	after H_2O, 15 min	10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS					QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.48479	13.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	mepiquat D_3	None	128.07 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
49	x	No	> 2 y	1.73	56.9	0.01	5	cold	Yes	after H_2O, 5 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	ILIS	None	99 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
58		Yes	> 2 y	0.012	-3.3	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
34		Yes	> 2 y	0.02	-2.8	0.02	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	EtOAc, 10 m	No		No	Dessication with Na_2SO_4, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	TPP	None	60 %	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789)
64	x	Yes	> 2 y	0.024	-2.6		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	57.1 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
24	x	No	> 2 y	0.0252	-2.5	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	80 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
97	x	No	> 2 y	0.025	-2.5	0.01	5	ambient	10 ml	No	mechanical shaking, 2 min	No	ACN, 10 mL ACN	No		No	Disp.-SPE (PSA/$MgSO_4$); as method	LC-MS/MS (QQQ), internal standard	MM-SL	TPP	None	75 % (0.01 mg/kg)	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM, EURL SRM
53		No	> 2 y	0.03	-2.2	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-SL	No	PrCal	86 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
37	x	Yes	> 2 y	0.034	-2.0	0.01	5	ambient	10 ml	after H_2O, 30 min	ultra turrax, 1 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	TMA	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
95		No	< 1 y	0.039	-1.7		5	ambient	10 ml	after H_2O, 15 min	10 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)
90		Yes	> 2 y	0.0414	-1.5	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	None	64 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
65	x	No	None	0.0461	-1.2	0.05	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	60 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.049	-1.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	64 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
55		Yes	> 2 y	0.05	-1.0	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Disp.-SPE (ODS/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	93 % (0.04 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
36		Yes	> 2 y	0.0523	-0.9	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
89		Yes	< 1 y	0.052	-0.9	0.005	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	73 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
2		Yes	> 2 y	0.053	-0.8	0.01	10	deep frozen	No	No	ASE, 15 min	No	ACN	No		No	aluminium oxide	LC-MS/MS (QQQ)	StAdd-SP	generic IS	PrCal	101 % (0.05 et 0.1 mg/kg)	SB-EUPT	2	Cf. extraction, clean up and chromatography
4	x	No	None	0.0532	-0.8	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	propamocarb D_7	StAdd-SP			1	A-QuEChERS (with 1 % FA)
16		Yes	> 2 y	0.0553	-0.7	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out; $CaCl_2$	LC-MS/MS (QQQ)	PS-ML	No	None	86.3 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	> 2 y	0.055	-0.7	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN, 10 ml	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	91 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003)
84	x	Yes	> 2 y	0.055	-0.7	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
113		No	> 2 y	0.055	-0.7	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	104 % (0.05 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
23	x	No	1 - 2 y	0.057	-0.6	0.01	5		10 ml	after H_2O, 30 min	1 min		ACN	No			Freezing out	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	86 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
32		Yes	> 2 y	0.056	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
35		No	> 2 y	0.056	-0.6	0.01	10	ambient	20 ml	No	mechanical shaking, 20 min	No	EtOAc	1× ($NaHCO_3$)		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-SL	pirimicarb-D_6	None	75 % (0.032 mg/kg)	SB-EUPT	1	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), Extraktion time
47	x	Yes	> 2 y	0.057	-0.6	0.01	5	ambient	10 ml	after H_2O, 30 min	manual shaking, 2 min	No	ACN				Freezing out	LC-MS/MS (QQQ)	MM-ML	No	None	77.5 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
98	x	Yes	1 - 2 y	0.056	-0.6	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		80 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
107		Yes	> 2 y	0.0571	-0.6	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
51	x	Yes	> 2 y	0.058	-0.5	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	TPP	StAdd-EA	80 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
60	x	No	> 2 y	0.0575	-0.5	0.01	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	carbaryl	PrCal	87 %	SB-EUPT	3	Modified QuEChERS
74	x	Yes	> 2 y	0.058	-0.5	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No	other				QuEChERS-Citrate buffered (EN 15662)
94	x	Yes	> 2 y	0.0583	-0.5	0.01	5	deep frozen	10 g	after H_2O and organic solvent, 5 min	manual shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	106 % (0.01 mg/kg)	SB-EUPT	5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
105		Yes	> 2 y	0.058	-0.5	0.01	5	ambient	10 ml	after H_2O, 60 min	manual shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	PrCal	80 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), dilution before LC-MS
11		Yes	> 2 y	0.06	-0.4	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	pirimicarb-D_6		88 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
76		Yes	> 2 y	0.06	-0.4	0.01	5	ambient	Yes	after H_2O and organic solvent, 5 min			ACN	No			Disp.-SPE (PSA/$MgSO_4$)	LC-Orbitrap	PS-ML	atrazine	None	89 %			QuEChERS-Citrate buffered (EN 15662)
109		Yes	> 2 y	0.06	-0.4	0.01	2	slightly frozen	10 ml	No	manual shaking, 2 min		ACN	No			Freezing out, Disp.-SPE (PSA/$MgSO_4$)	GC-MSD	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)
111		Yes	> 2 y	0.06	-0.4	0.01	5	just thawed	Yes	after H_2O, 30 min	mechanical shaking, > 60 min	No	H_2O/MeOH			No	Centrifugation, Filtration	LC-MS/MS (QQQ), two transitions	MM-ML	TBP	None	91 % (2x MRRL)	SB-EUPT	1	other
33		Yes	> 2 y	0.062	-0.3	0.01	5	ambient	5 ml	No	ultra turrax, 2 min	No	MeOH + 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	TPP					in house method
49	x	Yes	> 2 y	0.061	-0.3	0.01	5	cold	Yes	after H_2O, min	manual shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-SL	No	None	80 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
62		Yes	> 2 y	0.062	-0.3	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No		71 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
25		Yes	> 2 y	0.064	-0.2	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ)	MM-ML	TPP	other	78 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
63		Yes	> 2 y	0.064	-0.2	0.02	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	40 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
78		Yes	> 2 y	0.065	-0.1	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	ACN	No		No	Centrifugation, Dessication with Na_2SO_4	LC-MS/MS (QQQ)	MM-ML	generic IS	None	83 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003)
31		No	> 2 y	0.067	0.0	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	120 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
43		No	> 2 y	0.067	0.0	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
81	x	Yes	> 2 y	0.0658	0.0	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	88 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
85		No	< 1 y	0.067	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		54 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
12	x	Yes	1 - 2 y	0.068	0.1	0.01	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	StAdd-SP	daminozide D_6	StAdd-SP	91.4 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
19		Yes	1 - 2 y	0.068	0.1	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	103 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
106		Yes	> 2 y	0.069	0.1	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	81 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
40		Yes	< 1 y	0.07	0.2	0.01	5	slightly frozen	Yes	after H_2O, 10 min	mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	No	PrCal				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
45		Yes	> 2 y	0.0696	0.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 20 min	mechanical shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA), 10 ml (MeOH 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI +	MM-ML	No	None	101 % (0.1 mg/kg spiking level)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
46		Yes	> 2 y	0.0704	0.2	0.005	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	TPP	None	100 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
15		Yes	> 2 y	0.0708	0.3	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	TPP	StAdd-EA	75 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
27		Yes	> 2 y	0.072	0.3	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	75 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
38		Yes	> 2 y	0.071	0.3	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	81.6 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
44	x	Yes	> 2 y	0.0718	0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
56		Yes	> 2 y	0.0718	0.3	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Disp.-SPE (PSA/ $MgSO_4$), Filtration	LC-MS/MS (QQQ)	MM-ML	No	PrCal	45 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
72		Yes	1 - 2 y	0.0717	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	105 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
77		Yes	> 2 y	0.071	0.3	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
82		Yes	> 2 y	0.072	0.3	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1×		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ), ESI+	MM-ML	No	RecF	29 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662); Unusal low recovery, reason is not clear
21		Yes	None	0.074	0.4	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	94 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
54	x	Yes	> 2 y	0.073	0.4	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	94 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66
114		Yes		0.027	0.4	0.01														generic IS	StAdd-SP	96 %	SB-EUPT	1	no data
5		Yes	> 2 y	0.075	0.5	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
70	x	Yes	> 2 y	0.0756	0.5	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI pos	MM-ML	ILIS	None	100 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
91	x	Yes	> 2 y	0.0756	0.5	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	81.9 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
6	x	Yes	> 2 y	0.076	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
30		Yes	> 2 y	0.077	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Centrifugation	LC-MS/MS (QQQ), 189/74	MM-ML	No	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
41	x	No	None	0.0769	0.6	0.01	5	ambient	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	70 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
75		Yes	> 2 y	0.077	0.6	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	StAdd-EA	propamocarb D_7	StAdd-EA	65 %	QC	> 5	QuEChERS-Citrate buffered (EN 15662), additional with QuPPe-Method
87		Yes	> 2 y	0.077	0.6	0.01	5	just thawed	9.5 g	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	No		No	Liq-liq part.; ChemElut	LC-MS/MS (QQQ), ESI pos.	MM-ML	carbendazim-D_4	other	93.6 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut; Dilution of extract 1:50
10		Yes	> 2 y	0.0787	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	87 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
73		Yes	> 2 y	0.078	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	104 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
61		Yes	> 2 y	0.08	0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	81 %	QC		QuEChERS-Citrate buffered (EN 15662)
103		Yes	1 - 2 y	0.08	0.8	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			Disp.-SPE; PSA-C18	LC-MS/MS (QQQ)	PS-ML	triclabendazole	RecF	31 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), Purification
1		No	> 2 y	0.081	0.9	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	47 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
18		Yes	> 2 y	0.081	0.9	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.0816	0.9	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Isooctane			No		LC-MS/MS (QQQ)		No	StAdd-SP	80 %	SB-EUPT	2	MeOH extraction
80		Yes	> 2 y	0.082	0.9	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.08399	1.0	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	127.7 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Propamocarb (Assigned value = 0.067 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
83		Yes	> 2 y	0.085	1.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 20 min	No	ACN	1× (buffered pH = 5)		No	Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ), ESI positive	StAdd-SP	No	PrCal				QuEChERS-Citrate buffered (EN 15662)
9		Yes	> 2 y	0.087	1.2	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	No	Isooctane				SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	79 % (not corrected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
26	x	No	> 2 y	0.086	1.2		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	86.9 %		3	QuEChERS - Original Version (J. AOAC 86, 2003)
118		No	None	0.0884	1.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	TPP	None			4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
22		Yes	> 2 y	0.0892	1.4	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered, pH 4; PSA/$MgSO_4$, pH > 8)		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI positiv	MM-ML	pirimicarb-D_6, TDCPP	None	109 % (0.05 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
79		No	< 1 y	0.092	1.5	0.01	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	98 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
8		Yes	> 2 y	0.096	1.8	0.01	10	ambient	20 ml	after H_2O and	ultra turrax		Isooctane				No	LC-MS/MS (QQQ)		ILIS	other	78 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
3		Yes	> 2 y	0.098	1.9	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	90 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	> 2 y	0.101	2.1	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	ILIS	PrCal	90 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
71		No	None	0.115	2.9	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	RecF	61.2 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
67	x	Yes	> 2 y	0.1237	3.4	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	PS-ML	ILIS		90 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
116		Yes	> 2 y	0.15	5.0	0.02	2	ambient	No	No	mechanical shaking, 5 min	No	ACN, H_2O	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	102 %	SB-EUPT	1	other (extraction with ACN:H_2O)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
115		Yes	None	0.02	-3.2		10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	labelled	None	100 %		> 5	QuEChERS-Citrate buffered (EN 15662), modified method
114		No		0.029	-2.8	0.01														generic IS	StAdd-SP	100 %	SB-EUPT	1	no data
118		No	None	0.0308	-2.7		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	TPP	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
24	x	No	> 2 y	0.0338	-2.6	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	89 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
64	x	Yes	> 2 y	0.038	-2.4	0.05	15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	31.9 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
85		No	< 1 y	0.044	-2.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		26 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
34		Yes	> 2 y	0.049	-2.0	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	No	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	111 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
58		Yes	> 2 y	0.068	-1.2	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
98	x	Yes	1 - 2 y	0.068	-1.2	0.01	5	deep frozen	Yes	No	mechanical shaking, 2 min		ACN				Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	No		93 %	SB-EUPT	> 5	QuEChERS-Citrate buffered (EN 15662)
16		Yes	> 2 y	0.071	-1.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	83 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
31		No	1 - 2 y	0.07	-1.1	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	78 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
18		Yes	> 2 y	0.073	-1.0	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
20		Yes	> 2 y	0.075	-0.9	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	70 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
36		Yes	> 2 y	0.0754	-0.9	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
70	x	No	None	0.0806	-0.7	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	95 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
103		No	None	0.081	-0.7	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	No	None	79 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
8		Yes	> 2 y	0.082	-0.6	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	87 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
30		Yes	> 2 y	0.084	-0.6	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabutyl-ammoniumhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:112/154/133	PS-SL	No	None	85 %	SB-EUPT	1	other (with methylation)
67	x	Yes	> 2 y	0.0834	-0.6	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		90 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
71		No	None	0.083	-0.6	0.01	5	cold	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN / 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	73.7 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
65	x	No	< 1 y	0.0898	-0.3	0.02	5	ambient	10 ml	after H_2O, 5 min	ultrasonic bath, 20 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	68 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	1 - 2 y	0.0893	-0.3	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	100 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
87		Yes	> 2 y	0.09	-0.3	0.01	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	77.8 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
116		No	None	0.09	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	86 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
4	x	No	None	0.0917	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
6	x	Yes	> 2 y	0.094	-0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
38		Yes	> 2 y	0.092	-0.2	0.01	5	ambient	10 ml	No	manual shaking, 2 min	No	ACN	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	95.8 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
40		Yes	> 2 y	0.093	-0.2	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifugation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	84 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
78		Yes	> 2 y	0.092	-0.2	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifugation	LC-MS/MS (QQQ)	PS-ML	generic IS	None	83 %	SB-EUPT	1	in house method
84	x	No	None	0.093	-0.2	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
89		Yes	< 1 y	0.094	-0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
91	x	No	< 1 y	0.0921	-0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	93.7 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
15		Yes	> 2 y	0.0958	-0.1	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	83 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
56		Yes	> 2 y	0.0947	-0.1	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	101 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
79		No	< 1 y	0.096	-0.1	0.01	5	cold	Yes	after H_2O, 20 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	84 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
61		Yes	> 2 y	0.097	0.0	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	98 %	QC		QuEChERS-Citrate buffered (EN 15662)
73		No	None	0.0989	0.0	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	100.7 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
117		Yes	1 - 2 y	0.097	0.0	0.01	2	just thawed	Yes	after H_2O, min	manual shaking		ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	97 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
2		Yes	> 2 y	0.1	0.1	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column	LC-MS/MS (QQQ)	StAdd-SP	MCPP-D_3	PrCal	92 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
10		Yes	> 2 y	0.1	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
11		No	< 1 y	0.099	0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		101 % (0.02 and 0.1 mg/kg)	SB-EUPT	2	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
19		Yes	1 - 2 y	0.1	0.1	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
26	x	No	1 - 2 y	0.101	0.1		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	76.8 %		3	QuEChERS-Citrate buffered (EN 15662)
28		No	> 2 y	0.1	0.1	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	99 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
32		No	None	0.101	0.1	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
53		No	> 2 y	0.1	0.1	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	101 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
107		Yes	> 2 y	0.101	0.1	0.01	5	deep frozen	Yes	after H_2O and organic solvent, 5 min	manual shaking, 1 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	95 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
25		Yes	1 - 2 y	0.103	0.2	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	114 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
55		Yes	> 2 y	0.102	0.2	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	bentazone-D_6	PrCal	94 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
74	x	Yes	> 2 y	0.103	0.2	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
80		Yes	> 2 y	0.103	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
83		Yes	> 2 y	0.102	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
3		Yes	> 2 y	0.104	0.3	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	110 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
51	x	Yes	> 2 y	0.105	0.3	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	ILIS	StAdd-EA	113 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
54	x	Yes	> 2 y	0.105	0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	ultra turrax, 1 min	No	Isooctane, H_2O, NH_4OAc	No		No	Filtration	LC-MS/MS (QQQ), Quattro 1er	MM-ML	oxfendazole	None	93 %	SB-EUPT	1	Hanot et al, JChromA 2015;1384;53-66
72		Yes	1 - 2 y	0.105	0.3	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	89 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
95		No	< 1 y	0.106	0.3		5	ambient	10 ml	after H_2O, 15 min	10 min	No	ACN	No		No	Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No					QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bentazone (Assigned value = 0.098 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
45		Yes	> 2 y	0.107	0.4	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	99 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
21		Yes	> 2 y	0.11	0.5	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	117 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
43		Yes	> 2 y	0.109	0.5	0.01	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
106		Yes	> 2 y	0.11	0.5	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	102 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
46		Yes	1 - 2 y	0.112	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	bentazone D_7	None	99 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
44	x	Yes	1 - 2 y	0.115	0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN			No	Freezing out	LC-MS/MS (QQQ)		No	StAdd-SP				A-QuEChERS (with 1 % FA)
57		Yes	> 2 y	0.116	0.7	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	119 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
82		Yes	> 2 y	0.116	0.7	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	59 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	0.116	0.7	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	95 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
12	x	No	None	0.118	0.8	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	109.5 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
5		Yes	> 2 y	0.12	0.9	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
75		Yes	> 2 y	0.12	0.9	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	109 %	QC	> 5	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.125	1.1	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
33		Yes	> 2 y	0.126	1.2	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
60	x	No	< 1 y	0.134	1.5	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/ $MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	113 %	SB-EUPT	3	Modified QuEChERS
63		Yes	1 - 2 y	0.14	1.7	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	50 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
27		Yes	> 2 y	0.16	2.5	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	93 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.17459	3.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	130.05 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
113		No			-3.7 (FN)		5	ambient										LC-MS/MS (QQQ)							A-QuEChERS (with 1 % FA)
115		Yes			-3.7 (FN)	0.01	10	deep frozen	No	No	mechanical shaking, 20 min	No	ACN			No	Centrifugation, Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	PS-ML					QuEChERS-Citrate buffered (EN 15662), modified method
34		Yes	> 2 y	0.039	-2.8	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	No	LC-MS/MS (QQQ), ESI neg	MM-ML	No	None	71 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
114		Yes		0.043	-2.6	0.02														generic IS	StAdd-SP	98 %	SB-EUPT	1	no data
118		No	None	0.0521	-2.3		2	ambient	Yes	after H_2O, 10 min		No	Isooctane	No			Centrifugation	LC-MS/MS (QQQ), HESI, positive	PS-ML	nicarbazin	None			2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
24	x	No	> 2 y	0.0579	-2.2	0.01	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 5 min	No	EtOAc	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	92 %	QC	> 5	SweEt type (T. Pihlström et al. Anal. Bioanal. Chem (2003, 89, 1773-1789), No HOAc added to the extraction solvent.
36		Yes	> 2 y	0.0636	-2.0	0.01	3	ambient	7.5 ml	No	mechanical shaking, 30 min	No	H_2O, 10 ml ACN + 1 % HAc	No		No	Liq-liq part., Filtration	LC-MS/MS (QQQ), 2 transitions	StAdd-EA	No	StAdd-EA	std add.l: 100 µg/kg		1	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), No dSPE clean up
64	x	Yes	> 2 y	0.062	-2.0		15	deep frozen	15 ml	after H_2O, 20 min		No	Acetone, DCM, Acetone, PE	No		No	Filtration	LC-MS/MS (QQQ)	PS-ML	No	None	39.6 %	SB-EUPT	1	Mini-Luke-Type (Acetone DCM-PE), Dilution Step
20		Yes	> 2 y	0.0805	-1.4	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	103 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
85		No	< 1 y	0.08	-1.4	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP		102 % (0.01 mg/kg)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662), Only first estraction
18		Yes	> 2 y	0.083	-1.3	0.01	5	ambient	10 ml.		5 min		ACN	No			No	LC-MS/MS (QQQ)	MM-ML	generic IS		90 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
58		No	> 2 y	0.091	-1.1	0.01	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
30		Yes	> 2 y	0.1	-0.8	0.01	10	ambient	80 mL	after H_2O, 5 min	ultra turrax, 2 min	alkaline with NaOH	Acetone, CyH, EtOAc	1×		Methylation with tetrabu-tyl-ammoni-umhydroxide/ iodomethane		GC-MSD (following derivatization), m/z:91/276/289	PS-SL	No	None	91 %	SB-EUPT	1	other (with methylation)
61		Yes	> 2 y	0.1	-0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	99 %	QC		QuEChERS-Citrate buffered (EN 15662)
78		Yes	> 2 y	0.099	-0.8	0.01	10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	No		No	Dessication with Na_2SO_4, Centrifuga-tion	LC-MS/MS (QQQ)	PS-ML	generic IS	None	81 %	SB-EUPT	2	in house method
103		Yes	1 - 2 y	0.104	-0.7	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	No	None	90 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
32		No	None	0.105	-0.6	0.01	5	just thawed	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	1× (1 % FA in ACN)		No	Filtration	LC-MS/MS (QQQ), API 4000 MRM	StAdd-EA	No	StAdd-EA				A-QuEChERS (with 1 % FA)
87		Yes	> 2 y	0.108	-0.6	0.02	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Liq-liq part.; ChemElut pH 4.8	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	66.9 %	SB-EUPT	1	Klein, Alder, J. AOAC 86/1015/2003, ChemElut pH 4.8; Dilution of extract 1:50
117		Yes	1 - 2 y	0.108	-0.6	0.01	2	just thawed	Yes	after H_2O, min			ACN					LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	113 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
8		Yes	> 2 y	0.111	-0.5	0.01	10	ambient	20 ml	after H_2O and organic solvent, 5 min	ultra turrax		ACN				No	LC-MS/MS (QQQ)		ILIS	other	95 %	SB-EUPT	2	0-tins: QuEChERS-based mth by EURL-SRM

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
2		Yes	> 2 y	0.114	-0.4	0.01	20	deep frozen	No	after H_2O and organic solvent, > 360 min	mechanical shaking, 30 min	No	acetone / phosphate buffer	1× (NaOH pH 8)		No	SPE-column	LC-MS/MS (QQQ)	StAdd-SP	MCPP-D_3	PrCal	117 % (0.1 mg/kg)	SB-EUPT	1	Cf. extraction, clean up and chromatography
80		Yes	> 2 y	0.113	-0.4	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ), API 4000, scheduled MRM	MM-ML	No		70 %	SB-EUPT		QuEChERS-Citrate buffered (EN 15662)
57		Yes	> 2 y	0.116	-0.3	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		ACN				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	102 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
67	x	Yes	> 2 y	0.1161	-0.3	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		78 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
73		No	None	0.116	-0.3	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	90 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
89		Yes	< 1 y	0.115	-0.3	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 10 min		ACN					LC-MS/MS (QQQ)	MM-ML	No	PrCal	60 %	SB-EUPT	2	QuEChERS - Original Version (J. AOAC 86, 2003), without cleanup
40		Yes	> 2 y	0.118	-0.2	0.01	5	slightly frozen	Yes	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE, Centrifugation, QuEChERS salts without PSA	LC-Orbitrap, Q-Exactive	MM-ML	mecoprop D_3	None	96 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
53		No	> 2 y	0.118	-0.2	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	99 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
56		Yes	> 2 y	0.12	-0.2	0.01	5		10 ml	after H_2O, 30 min	mechanical shaking, 1 min	No	ACN	No			Filtration	LC-MS/MS (QQQ)	MM-ML	No	None	102 % (Spiking at LOQ)	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
75		Yes	> 2 y	0.12	-0.2	0.01	5	ambient	10 ml	after H_2O and organic solvent, 15 min	manual shaking, 1 min	No	ACN with 1 % FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	97 %	QC	> 5	A-QuEChERS (with 1 % FA)
81	x	Yes	> 2 y	0.12	-0.2	0.01	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	93 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
11		Yes	> 2 y	0.121	-0.1	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	NaCl, $MgSO_4$	LC-Orbitrap	MM-ML	nicarbazin		95 % (0.02 and 0.1 mg/kg)	SB-EUPT	4	QuEChERS - Acetate buffered (AOAC Official Method 2007.01)
15		Yes	> 2 y	0.123	-0.1	0.002	5	ambient	10 ml	after H_2O, 20 min	manual shaking, 3 min	No	ACN	1× (addition of FA)		No	No	LC-MS/MS (QQQ), 2 transitions	MM-ML	nicarbazin	StAdd-EA	87 % (0.040 mg/kg)	SB-EUPT	3	QuEChERS - Original Version (J. AOAC 86, 2003)
84	x	Yes	> 2 y	0.122	-0.1	0.01	5	slightly frozen	Yes	No	manual shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	91 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
3		Yes	> 2 y	0.125	0.0	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
66		Yes	1 - 2 y	0.126	0.0	0.01	5	cold	10 ml	after H_2O, 15 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-Orbitrap	MM-ML	No	PrCal	100 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
70	x	No	None	0.124	0.0	0.01	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	No	RecF	60 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
91	x	Yes	> 2 y	0.124	0.0	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	92.6 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
21		Yes	> 2 y	0.127	0.1	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
22		Yes	> 2 y	0.129	0.1	0.01	5	ambient	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	2× (citrate buffered (pH 4), PSA/$MgSO_4$ (pH > 8))		No	Disp.-SPE (PSA/$MgSO_4$), Freezing out	LC-MS/MS (QQQ), ESI negativ	MM-ML	nicarbazin	None	99 % (0.1 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
45		Yes	> 2 y	0.127	0.1	0.01	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	96 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
46		Yes	1 - 2 y	0.127	0.1	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN w.1 %FA	No		No	Freezing out	LC-MS/MS (QQQ)	MM-ML	nicarbazin	None	94 %	SB-EUPT	2	A-QuEChERS (with 1 % FA)
51	x	Yes	> 2 y	0.127	0.1	0.01	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	111 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.128	0.1	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
4	x	No	None	0.133	0.2	0.01	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
10		Yes	> 2 y	0.133	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	97 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
12	x	Yes	1 - 2 y	0.13	0.2	0.01	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	102.3 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
29	x	Yes	> 2 y	0.13	0.2	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	84 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
43		Yes	> 2 y	0.132	0.2	0.003	5	ambient	Yes	No	mechanical shaking, 15 min	No	ACN	1× (citrate buffer)		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	None				QuEChERS-Citrate buffered (EN 15662)
63		Yes	1 - 2 y	0.13	0.2	0.01	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		ACN				Centrifugation, Filtration	LC-MS/MS (QQQ)		other compound	StAdd-SP	45 %	SB-EUPT		QuEChERS - Original Version (J. AOAC 86, 2003)
83		Yes	> 2 y	0.132	0.2	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	Yes	ACN	2×		No	phase separation after QuEChERS salt addition	LC-MS/MS (QQQ), ESI negative	StAdd-SP	No	PrCal				Quechers modular L00.00 115/1 E6-C0-D1-Q7
72		Yes	1 - 2 y	0.135	0.3	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	94 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
6	x	Yes	> 2 y	0.139	0.4	0.01	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
9		Yes	1 - 2 y	0.137	0.4	0.01	10	ambient	9 ml	after H_2O, 30 min	ultra turrax, 60 min	20 % NaOH; pH 12	Isooctane	1× (3-4)			SPE-column; Extrelut	LC-MS/MS (QQQ)	MM-ML	No	PrCal	80 % (not corrected)	SB-EUPT	3	Klein, Alder, J. AOAC 86/1015/2003
55		Yes	> 2 y	0.138	0.4	0.01	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	bentazone-D_6	PrCal	104 % (0.04 mg/kg)	SB-EUPT	1	A-QuEChERS (with 1 % FA)
31		No	1 - 2 y	0.14	0.5	< 0.01	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	TPP, Diuron D_6, pirimicarb D_6	None	80 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662), w/o Step 2 and from Step 1 directly 0.4 ml to a vial with 0.6 ml MeOH/H_2O
106		Yes	> 2 y	0.14	0.5	0.01	5	deep frozen	Yes		2 min	No						LC-MS/MS (QQQ)	MM-ML	other compound	None	100 %	SB-EUPT	1	QuEChERS - Original Version (J. AOAC 86, 2003), without PSA
5		Yes	> 2 y	0.147	0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Bromoxynil (Assigned value = 0.125 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
23	x	Yes	1 - 2 y	0.146	0.7	0.01	5		10 ml	after H_2O, 30 min	1 min	Yes	ACN	No			Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	PS-ML	other compound	StAdd-SP	113 %	SB-EUPT	3	other
26	x	No	1 - 2 y	0.147	0.7		5	ambient	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1×			No	LC-MS/MS (QQQ)	MM-SL	No	StAdd-EA	77.3 %		3	QuEChERS-Citrate buffered (EN 15662)
27		Yes	> 2 y	0.148	0.7	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	102 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
28		No	1 - 2 y	0.146	0.7	0.01	5	cold	4 ml	after H_2O, 5 min	mechanical shaking, 10 min		ACN, 10 ml					LC-MS/MS (QQQ)	MM-ML	(4-chloro-3.5-di-methyl-phenoxy) HOAc	None	86 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
74	x	No	None	0.146	0.7	0.01	5	cold	Yes	No	manual shaking, 2 min	No	ACN	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				Quechers without PSA clean up
19		Yes	1 - 2 y	0.15	0.8	0.02	5	ambient	10 ml	after H_2O, 10 min	5 min	300 µl 5N NaOH, 30 min, 300 µl 5N H_2SO_4	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	115 % (0.020 and 0.200 mg/kg)	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
25		Yes	> 2 y	0.151	0.8	0.01	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN					LC-MS/MS (QQQ), ESI neg.	MM-ML	nicarbazin	other	128 % (0.05 mg/kg)	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), no PSA clean-up
33		Yes	> 2 y	0.154	0.9	0.01	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	nicarbazin					QuEChERS - Original Version (J. AOAC 86, 2003)
82		Yes	> 2 y	0.153	0.9	0.01	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	66 % (0.1 mg/kg)	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
1		No	> 2 y	0.155	1.0	0.01	2	deep frozen	10 ml	after H_2O, 30 min	ultrasonic bath, 15 min	No	ACN	No		No	Freezing out, Disp.-SPE (PSA/$MgSO_4$)	LC-MS/MS (QQQ)	MM-ML	No	None	112 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
60	x	No	> 2 y	0.173	1.5	0.02	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	ACN	No		No	Disp.-SPE (ODS/$MgSO_4$), Freezing out	LC-MS/MS (QQQ)	MM-ML	2,4-D	PrCal	101 %	SB-EUPT	3	Modified QuEChERS
100		Yes	> 2 y	0.20976	2.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	135.22 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol

deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

N-Acetyl glufosinate (Assigned value = 0.319 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
60	x	No			-3.7 (FN)	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			Centrifugation	no data	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
74	x	No	None	0.26	-0.7	0.05	5	cold	Yes	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	MM-ML	No	other				QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
12	x	Yes	> 2 y	0.279	-0.5	0.05	5	just thawed	10 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Filtration	LC-MS/MS (QQQ)	MM-ML	N-acetyl glufosinate D_3	StAdd-EA	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
14		No	None	0.278	-0.5	0.02	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML	No	StAdd-EA	83 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	No	> 2 y	0.282	-0.5	0.02	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	N-acetyl glufosinate-D_3 disodium salt	None	86 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
6	x	No	< 1 y	0.305	-0.2	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	< 1 y	0.306	-0.2	0.05	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)			No		LC-MS/MS (QQQ)	MM-ML	glyphosate 1,2-$^{13}C_2$ ^{15}N	PrCal	83 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
15		No	None	0.321	0.0	0.02	5	ambient	10 ml	after H_2O, 10 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (addition of FA)		No	Centrifugation, Filtration	LC-MS/MS (QQQ), 2 transitions	MM-ML	No	RecF	93 % (0.2 mg/kg)	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.318	0.0	0.01	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		glufosinate D_3	StAdd-SP	90 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
2		Yes	> 2 y	0.329	0.1	0.02	3	deep frozen	9.75 mL	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	PS-ML	No		101 % (0.16 mg/kg)	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Cf. extraction, clean up and chromatography
4	x	Yes	1 - 2 y	0.337	0.2	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	glufosinate D_3	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
21		No	None	0.335	0.2	0.02	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	91 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
18		No	1 - 2 y	0.35	0.4	0.02	5	ambient	10 ml.		20 min		Isooctane	No			Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS		95 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	1 - 2 y	0.35	0.4	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	101 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	None	0.356	0.5	0.1	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
51	x	No	< 1 y	0.416	1.2	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 1 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out	LC-MS/MS (QQQ)	StAdd-EA	glyphosate 2-^{13}C	StAdd-EA	113 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- # deep frozen (e.g. -18 °C), slightly frozen (e.g. -8 °C – -3 °C), just thawed (e.g. 0 °C – 3 °C), cold (e.g. 4 °C – 10 °C), cold (e.g. 4 °C – 10 °C)

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; StAdd-SP: Standard add. to sample portions); StAdd-EA: Standard add. to extract aliquots
2) ILIS : isotopically labelled internal standard
3) PrCal: Procedural calibration; StAdd-SP: Standard add. to sample portions; StAdd-EA: Standard add. to extract aliquots; RecF: Use a recovery factor
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Phosphonic acid (Assigned value = 0.584 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
14		No			-3.7 (FN)	0.05	5	deep frozen	10 ml	after H_2O, 20 min	mechanical shaking, 45 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Freezing out, Centrifugation, filtration	LC-MS/MS (QQQ)	MM-ML						QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
60	x	No	1 - 2 y	0.226	-2.5	0.04	5	cold	10 ml	after H_2O and organic solvent, 10 min	manual shaking, 2 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	PrCal	75 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
57		No	1 - 2 y	0.276	-2.1	0.01	5	ambient	Yes	after H_2O, 5 min	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	StAdd-SP	86 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
25		Yes	1 - 2 y	0.334	-1.7	0.1	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)					LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	118 % (0.05 mg/kg)	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
100		Yes	> 2 y	0.35534	-1.6	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 5 min	No	MeOH + 1 % FA	No		No	No	LC-MS/MS (QQQ)	MM-ML	dietilfosfato-ac. fosfoforico	None	139.06 %	SB-EUPT	4	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
53		No	> 2 y	0.469	-0.8	0.05	5	deep frozen	10 ml		manual shaking, 1 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)			No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	74 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
87		No	< 1 y	0.534	-0.3	0.3	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	phosphonic acid $^{18}O_3$	other	93.6 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:20
8		No	1 - 2 y	0.553	-0.2	0.1	1	ambient	9 ml	after H_2O and organic solvent, 5 min	ultra turrax		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)		ILIS	other	89 %	SB-EUPT	2	O-tins: QuEChERS-based mth by EURL-SRM
21		Yes	1 - 2 y	0.559	-0.2	0.01	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	ILIS	StAdd-SP	82 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), QuPPe for plant origin
46		No	None	0.55	-0.2	0.05	5	ambient	10 ml	No	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	phosphonic acid $^{18}O_3$	None	98 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
61		Yes	1 - 2 y	0.56	-0.2	0.01	5	cold	No		mechanical shaking, 10 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No			No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	111 %	QC		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
82		Yes	1 - 2 y	0.566	-0.1	0.1	5	ambient	10 ml	No	manual shaking, 5 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	ILIS	None			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
20		Yes	1 - 2 y	0.585	0.0	0.1	10	ambient	15 ml	after H_2O, 120 min	ultra turrax, 1 min	No	H_2O, Acified MeOH	No		No		LC-MS/MS (QQQ)		phosphonic acid $^{18}O_3$	StAdd-SP	94 %	SB-EUPT	2	Acified MeOH/H_2O extraction; Accreditation in request
30		Yes	> 2 y	0.591	0.0	0.1	5	ambient	10 ml	after H_2O and organic solvent, 10 min	mechanical shaking, 15 min	No	Isooctane	No		No	Centrifugation	LC-MS/MS (QQQ), 81/79	PS-ML	$^{18}O_3$ phosphonic acid	PrCal	80 % (83 % O_3 phosphonic acid recovery)	SB-EUPT	1	in house method, QuPPe-based
6	x	No	< 1 y	0.598	0.1	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	No	LC-MS/MS (QQQ)	MM-ML	ILIS	PrCal	100 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides); no ISTD used for reported result
27		Yes	None	0.606	0.1	0.1	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				No	LC-MS/MS (QQQ)	StAdd-SP	No	StAdd-SP	73 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
58		No	None	0.592	0.1	0.05	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), Dilution
4	x	Yes	< 1 y	0.615	0.2	0.05	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	StAdd-SP	phosphonic acid $^{18}O_3$	StAdd-SP			1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)

- Abb. of solvent: ACN: acetonitrile; CyH: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
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Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

Phosphonic acid (Assigned value = 0.584 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach [1]	ISTD used [2]	Other means of correcting for recovery or matrix-effects? [3]	Recovery % (compound. level)	Recovery obtained from [4]	Recovery replicates considered	Method details / Comments
10		Yes	< 1 y	0.617	0.2	0.05	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	1× (1 % FA in MeOH)		No	No	LC-MS/MS (QQQ)	MM-ML	$^{18}O_4$ phosphonic acid	None	95 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
70	x	No	None	0.672	0.6	0.1	5	ambient	Total H_2O content 10 g	after H_2O, 15 min	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI neg	MM-ML	ILIS	None	88 %	SB-EUPT	> 5	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
81	x	No	None	0.717	0.9	0.05	5	cold	9.5 ml	No	mechanical shaking, 30 min	No	QuPPe solvent (+ H_2O, MeOH w. 1 % FA)	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	phosphonic acid $^{18}O_3$	None	100 %	SB-EUPT	2	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
78		Yes	< 1 y	0.76	1.2		10	ambient	Yes	after H_2O, 10 min	ultra turrax, 1 min	No	Isooctane	1×		No	Centrifugation	LC-MS/MS (QQQ)	PS-ML	ILIS	None	114 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
33		Yes	1 - 2 y	0.774	1.3	0.05	5	ambient	5 ml	No	mechanical shaking, 30 min	No	H_2O, MeOH 1 % FA	No		No	Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	phosphonic acid $^{18}O_3$					in house method
5		Yes	> 2 y	0.971	2.6	0.05	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	Isooctane	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	ILIS	PrCal	95 %	SB-EUPT	3	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides)
63		Yes	< 1 y	1.106	3.6	0.05	5	cold	10 ml	after H_2O, 10 min	manual shaking, 10 min		QuPPe solvent (+ H_2O, MeOH w. 1 % FA)				Centrifugation, Filtration	LC-MS/MS (QQQ)		ILIS	PrCal	60 %	SB-EUPT		QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), H_2O added in vial

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Supplement-2: Methods used by the participating laboratories (ordered by z-scores)

TFNG (Assigned value = 0.168 mg/kg)

Lab-Code SRM10-	NRL	within routine scope	Experience w. analysis of compound	Reported result [mg/kg]	z-Score	RL [mg/kg]	Sample weight [g]	Initial sample temperature #	Water addition	Soaking step	Extraction approach	Hydrolysis step?	Extraction- and/or partitioning solvents	pH modified?		Derivatisation	Cleanup	Determination technique	Calibration approach 1)	ISTD used 2)	Other means of correcting for recovery or matrix-effects? 3)	Recovery % (compound. level)	Recovery obtained from 4)	Recovery replicates considered	Method details / Comments
58		No	None	0.014	-3.7	0.02	5	deep frozen	Yes	after H_2O, 5 min	mechanical shaking, 20 min	No	EtOAc	No		No		LC-MS/MS (QQQ)	StAdd-EA	No	StAdd-EA			3	QuEChERS - Acetate buffered (AOAC Official Method 2007.01), Extraction with EtOAc and no dSPE
34		No	None	0.029	-3.3	0.01	5	deep frozen	10 ml	after H_2O, 5 min	mechanical shaking, 3 min	No	ACN, 10 ml	No		No	Disp.-SPE (PSA/$MgSO_4$), Centrifugation	LC-MS/MS (QQQ), ESI pos	MM-ML	TPP	None	95 %	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
87		No	< 1 y	0.102	-1.6	0.04	5	just thawed	9.5 g pH 4	after H_2O, 10 min	ultra turrax, 2 min	No	MeOH	1× (pH 4)		No	Centrifugation	LC-MS/MS (QQQ), ESI neg.	MM-ML	No	other	83 %	SB-EUPT	1	QuPPe for products of plant origin (EURL-SRM mth for polar pesticides), no acidified MeOH; Dilution of extract 1:50
12	x	No	None	0.132	-0.8	0.02	5	just thawed	10 ml	No	mechanical shaking, 15 min	No	ACN, acidified with 1 % FA	No		No	Freezing out, Centrifugation	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	86.5 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
81	x	Yes	< 1 y	0.134	-0.8	0.02	5	cold	10 ml	after H_2O, 30 min	manual shaking, 1 min	No	ACN	1× (citrate buffered)		No		LC-MS/MS (QQQ)	MM-ML	No	None	69 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
113		Yes	> 2 y	0.134	-0.8	0.01	5	ambient										LC-MS/MS (QQQ)	MM-ML	No	None	85 % (0.05 mg/kg)	SB-EUPT	3	A-QuEChERS (with 1 % FA)
30		Yes	> 2 y	0.14	-0.7	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 10 min	No	ACN	No		No	Disp.-SPE (PSA/$MgSO_4$), Centrifugation	LC-MS/MS (QQQ), 247/182	MM-ML	No		78 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
73		No	None	0.14	-0.7	0.01	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 5 min	No	ACN	1× (1 % FA)		No	No	LC-MS/MS (QQQ)	PS-ML	No	None	78.8 %	SB-EUPT	3	A-QuEChERS (with 1 % FA)
103		Yes	< 1 y	0.156	-0.3	0.01	10	ambient	Yes	after H_2O, 15 min	mechanical shaking, 5 min		ACN	No			No	LC-MS/MS (QQQ)	PS-ML	mecoprop D_3	RecF	50 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662), No purification
10		No	> 2 y	0.159	-0.2	0.02	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	1× (Citrate Buffer pH 5.5)		No	No	LC-MS/MS (QQQ)	MM-ML	MCPA D_6	None	83 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
67	x	No	None	0.16	-0.2	0.01	5	ambient	10 ml	after H_2O, 30 min	mechanical shaking, 10 min	No	ACN, 1 % FA	No		No		LC-MS/MS (QQQ)	MM-ML	No		79 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
77		Yes	> 2 y	0.16	-0.2	0.01												no data		No	StAdd-SP				QuEChERS - Original Version (J. AOAC 86, 2003)
21		No	None	0.164	-0.1	0.02	5	cold	10 ml	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP	84 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
29	x	Yes	< 1 y	0.165	-0.1	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN with 1 % FA	No		No	Centrifugation, Freezing out	LC-MS/MS (QQQ), Waters	MM-ML	No	None	75 % (0.05 and 0.1 mg/kg)	SB-EUPT	2	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
45		No	1 - 2 y	0.162	-0.1	0.02	5	cold	10 ml	No	mechanical shaking, 10 min	No	ACN, 10 ml	No		No	Centrifugation, Filtration	LC-MS/MS (QQQ), ESI -	MM-ML	No	None	95 % (0.1 mg/kg spiking level)	SB-EUPT	2	A-QuEChERS (with 1 % FA)
51	x	Yes	1 - 2 y	0.164	-0.1	0.02	5	slightly frozen	10 ml	after H_2O, 15 min	manual shaking, 15 min		ACN	No		No	Disp.-SPE, Freezing out, $MgSO_4$, NaCl	LC-MS/MS (QQQ)	MM-ML	nicarbazin	StAdd-EA	119 %	SB-EUPT	1	A-QuEChERS (with 1 % FA)
72		Yes	1 - 2 y	0.165	-0.1	0.02	5	ambient	10 ml	after H_2O and organic solvent, 15 min	mechanical shaking, 20 min	Yes	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	No	None	111 %	SB-EUPT	3	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
20		Yes	> 2 y	0.17	0.1	0.01	5	ambient	10 ml	after H_2O, 120 min	ultra turrax, 1 min	No	Acetone; DCM; PE	No		No		LC-MS/MS (QQQ)		No	StAdd-SP	101 %	SB-EUPT	2	Mini-Luke-Type (Acetone DCM-PE)
3		Yes	> 2 y	0.174	0.2	0.01	5	cold	10 ml	after H_2O and organic solvent, 5 min	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	None	93 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)

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TFNG (Assigned value = 0.168 mg/kg)

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25		Yes	< 1 y	0.174	0.2	0.05	5	deep frozen	10 ml	No	mechanical shaking, 20 min	No	ACN, 1 % FA	No				LC-MS/MS (QQQ)	MM-ML	nicarbazin	other	93 % (0.05 mg/kg)	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
33		Yes	1 - 2 y	0.18	0.3	0.02	10	ambient	10 ml	No	ultra turrax, 2 min	No	ACN	No		No	Centrifugation	LC-MS/MS (QQQ)	MM-ML	TPP					QuEChERS - Original Version (J. AOAC 86, 2003)
55		Yes	1 - 2 y	0.18	0.3	0.02	5	deep frozen	10 mL cold H_2O	after H_2O, 20 min	mechanical shaking, 20 min	No	ACN	No		No	Dessication with $MgSO_4$	LC-MS/MS (QQQ)	MM-ML	carbofuran-D_3	PrCal	92 % (0.04 mg/kg)	SB-EUPT	1	TFNA/TFNG: QuEChERS-based mth by EURL_SRM
27		Yes	> 2 y	0.186	0.4	0.01	2	ambient	Yes	after H_2O, 10 min	mechanical shaking, 15 min	No	ACN				Disp.-SPE (PSA/ $MgSO_4$)	LC-MS/MS (QQQ)	StAdd-SP	other compound	StAdd-SP	96 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)
91	x	No	< 1 y	0.188	0.5	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 5 min	No	ACN	No		No	No	LC-MS/MS (QQQ), 2 daughter ions	MM-ML	TPP	None	94.2 %	SB-EUPT	2	QuEChERS-Citrate buffered (EN 15662)
100		Yes	> 2 y	0.19125	0.6	0.01	5	ambient	10 ml	after H_2O, 5 min	mechanical shaking, 15 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	linuron D_6	None	122.41 %	SB-EUPT	4	QuEChERS-Citrate buffered (EN 15662)
4	x	No	None	0.197	0.7	0.02	5	slightly frozen	10 ml	No	mechanical shaking, 15 min		ACN	1× (1 % FA)		No	Freezing out	LC-MS/MS (QQQ)	StAdd-SP	nicarbazin	StAdd-SP			1	A-QuEChERS (with 1 % FA)
61		Yes	1 - 2 y	0.2	0.8	0.01	5	cold	5 ml	No	mechanical shaking, 2 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	88 %	QC		QuEChERS-Citrate buffered (EN 15662)
6	x	No	< 1 y	0.208	1.0	0.02	5	ambient	10 ml	after H_2O, 5 min	manual shaking, 1 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	MM-ML	No	PrCal	100 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
82		Yes	1 - 2 y	0.21	1.0	0.05	8	ambient	15 ml	No	mechanical shaking, 15 min	No	ACN	1× (citrate-buffered)		No	No	LC-MS/MS (QQQ), ESI-	MM-ML	No	RecF	40 % (0.1 mg/kg)	SB-other	4	QuEChERS-Citrate buffered (EN 15662); Unusal low recovery, reason is not clear
53		No	< 1 y	0.212	1.1	0.01	2	deep frozen	10 ml		mechanical shaking, 20 min	No	ACN			No	Disp.-SPE	LC-MS/MS (QQQ)	MM-SL	No	PrCal	109 %	SB-EUPT	1	QuEChERS-Citrate buffered (EN 15662)
5		Yes	> 2 y	0.217	1.2	0.02	5	ambient	10 ml	after H_2O, 15 min	mechanical shaking, 30 min	No	ACN	No		No	No	LC-MS/MS (QQQ)	StAdd-SP	TPP	PrCal	95 %	SB-EUPT	3	QuEChERS-Citrate buffered (EN 15662)

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