# EU PROFICIENCY TEST
# EUPT-SRM9, 2014

# Residues of Pesticides requiring Single Residue Methods

## Test Item:
## Whole Cow's Milk

## Final Report
### Supplementary Information on Analytical Methods

## Supplement-1: Methods used by the participating laboratories (ordered by Lab-Codes) | COMPULSORY ANALYTES

### 2,4-D (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | > 2 y* | 0.087 | 0.0 | 0.02 | 10 | cold | | 30 | ACN | No | No | | | LC-MS/MS QQQ | | MM-ML | Yes, nicarbazim | Yes-5 | | | | QuEChERS for acidic pesticides (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.083 | -0.2 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.0957 | 0.4 | 0.005 | 10 | cold | No | 15 | ACN | Yes, once, citrat buffer, pH 5 | No | Centrifugation, Freezing out, Filtration through syringe filter | | LC-MS/MS QQQ, ESI negativ | | MM-ML | Yes, nicarbazin | No | 104 % (0.2 mg/kg) | SB-EUPT | 3 | Analysis of Phenoxyalkanoic Acids in Milk using QuECgERS method an LC-MS/MS |
| 4 | | Yes | < 1 y# | 0.0875 | 0.0 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-Orbitrap | None | MM-ML | Yes, Mecoprop D3 | No | 98 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.0931 | 0.3 | 0.02 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.070 | -0.8 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | |
| 8 | | Yes | > 2 y | 0.107 | 0.9 | 0.02 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | Yes | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI neg | None | Std. add to extract aliquots | No | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | None | 0.0967 | 0.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | < 1 y | 0.072 | -0.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | > 2 y | 0.066 | -1.0 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | Yes | > 2 y | 0.103 | 0.7 | 0.01 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 117.1 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | > 2 y | 0.095 | 0.3 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.0821 | -0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 90 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.0366 | -2.3 | 0.01 | 10 | just thawed | No | 1 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ, Zorbax XDB-C18 | Std. add to extract aliquots | No | Yes-1 | 88.2 % (0.01 mg/kg) | SB-EUPT | 4 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 21 | | Yes | > 2 y | 0.105 | 0.8 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.084 | -0.2 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TMA | Yes-2 | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 23 | | Yes | 1 – 2 y | 0.068 | -0.9 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.0781 | -0.4 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 25 | | Yes | > 2 y‡ | 0.108 | 0.9 | 0.02 | 10 | cold | No | 10 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 108.5 % (0.04 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.091 | 0.2 | 0.01 | 25 | ambient | Yes, alkine with NaOH | > 60 | Acetone, CyHn, EtOAc first hydrolysis | Yes, once | Yes, with tetrabutyl-ammonium-hydroxid/ iosomethane | GPC, Gel-Permeation Chr/phy, acid/base distribution | | | GC-MSD, via second m/z | PS-SL | Yes, d3-Mecoprop, no calculation | No | | SB-EUPT | 1 | alkine hydrolysis extraction GPC, acid/base distribution, methykation,GC-MSD detection |
| 27 | | Yes | > 2 y | 0.071 | -0.8 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 28 | | Yes | > 2 y | 0.083 | -0.2 | 0.01 | 5 | ambient | | 10 | ACN | | | Filtration | | | LC-MS/MS QQQ | MM-SL | No | No | | | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 30 | | Yes | > 2 y | 0.113 | 1.2 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.079 | -0.4 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.088 | 0.0 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | > 2 y | 0.109 | 1.0 | | 5 | just thawed | | 10 | EtOAc | Yes, once, HOAc | | Dessication with $Na_2SO_4$, Z-Sep/C18 | | LC-MS/MS QQQ, ES- | LC-MS/MS QQQ, ES- | Std. add to sample portions | | Yes-2 | | SB-EUPT | 1 | |
| 37 | | No | > 2 y | 0.078 | -0.4 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 38 | x | Yes | 1 – 2 y | 0.0746 | -0.6 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.113 | 1.2 | 0.02 | 10 | | | 15 | ACN | No | | Freezing out, 2h | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazine | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 41 | x | Yes | > 2 y | 0.065 | -1.0 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 43 | | Yes | > 2 y | 0.0803 | -0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.101 | 0.6 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 46 | | No | > 2 y | 0.0961 | 0.4 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extraction solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, 'standard' C18 R.P. chromatography | None | MM-SL | No | No | | - | | Extraction with ACN+1 % FA. No partitioning step |
| 47 | x | No | None | 0.390 | 13.8 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 78 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | > 2 y | 0.082 | -0.3 | 0.02 | 5 | cold | No | 10 | ACN | No | No | None | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | | | |
| 49 | x | No | > 2 y | 0.088 | 0.0 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 50 | x | Yes | > 2 y | 0.0951 | 0.3 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | Yes-1 | | SB-EUPT | 1 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 51 | x | Yes | > 2 y | 0.101 | 0.6 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Dispersive-SPE (ODS/$MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-2 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 52 | x | No | > 2 y | 0.0881 | 0.0 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.0778 | -0.4 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | > 2 y | 0.088 | 0.0 | 0.01 | 5 | ambient | No | 20 | EtOAc | Yes, once | No | z-sep+ | | | None | MM-SL | Yes, Pirimicarb-D6 | Yes-1 | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |
| 55 | x | Yes | > 2 y | 0.080 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) Other, specify under "Solvent details" | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 56 | | Yes | > 2 y | 0.0744 | -0.6 | 0.002 | 10 | ambient | No | 15 | ACN | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI- | None | MM-ML | Yes, nicarbazim | No | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 58 | x | Yes | > 2 y | 0.071 | -0.8 | 0.02 | 15 | cold | No | 3 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Filtration | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 23.7 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS - Acetate buffered (AOAC Official Method 2007.01) |
| 59 | | Yes | > 2 y | 0.112 | 1.1 | 0.01 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation, 1.+150 mg ODS; 2. -15 °C | | | LC-MS/MS QQQ | MM-ML | Yes, 4Cl35DiMeAcedicA | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 60 | x | Yes | > 2 y | 0.081 | -0.3 | 0.02 | 10 | deep frozen | No, None | 10 | ACN | No, None | No, None | Freezing out | | LC-MS/MS QQQ, None | None | MM-SL | Yes, nicarbazin | No | 85.3 % (82 %; CV 3.5 %) | SB-EUPT | 3 | |
| 61 | | Yes | > 2 y | 0.0899 | 0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 63 | | Yes | > 2 y | 0.108 | 0.9 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, nicarbazin | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.0726 | -0.7 | 0.01 | 5 | cold | No | 1 | ACN | Yes, once, 1 % HOAc in extraction solvent | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS based |
| 66 | x | Yes | > 2 y | 0.189 | 4.6 | 0.02 | 10 | cold | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 3rd-33 | | Yes | > 2 y | 0.100 | 0.6 | 0.02 | 1 | ambient | No | 5 | ACN | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | PS-ML | No | No | 102 % (0.02 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-67 | | Yes | > 2 y | 0.077 | -0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 100 % (0.1 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-71 | | Yes | > 2 y | 0.070 | -0.8 | 0.01 | 5 | ambient | Yes, Evaporate to 1 mL | 20 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | Plant Protection Division Residue Analytical Method No. 68 |
| 3rd-72 | | Yes | 1 – 2 y | 0.070 | -0.8 | 0.01 | 10 | cold | No | 1 | ACN | Yes, once | No | Dispersive-SPE | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | 1 – 2 y | 0.326 | 0.6 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TPP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 2 | x | No | None | 0.543 | 3.7 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.291 | 0.1 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/$MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 98 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 5 | x | Yes | 1 – 2 y | 0.333 | 0.7 | 0.005 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.257 | -0.4 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 7 | x | No | 1 – 2 y | 0.327 | 0.6 | 0.01 | 10 | cold | No | 2 | Other, specify under "Solvent details" | Yes, once | No | Dispersive-SPE, C18/PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 8 | | Yes | 1 – 2 y | 0.277 | -0.1 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, QuEChERS citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |
| 9 | x | No | 1 – 2 y | 0.244 | -0.6 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.244 | -0.6 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.231 | -0.7 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | No | > 2 y | 0.354 | 1 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 109.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.325 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.296 | 0.2 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 95 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.5 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.274 | -0.1 | 0.1 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | No | 1 – 2 y | 0.110 | -2.4 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | No | 1 – 2 y | 0.313 | 0.4 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, addition of QuEChERS citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 25 | | Yes | 1 – 2 y‡ | 0.300 | 0.2 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 102.1 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | 1 – 2 y | 0.295 | 0.2 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation,only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | No | 1 – 2 y | 0.239 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | 1 – 2 y | 0.220 | -0.9 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.066 | -3.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | 1 – 2 y | 0.288 | 0.1 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 35 | | Yes | 1 – 2 y | 0.300 | 0.2 | 0.02 | 5 | ambient | No | 10 | $H_2O$,ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltri-methylammonium chloride | No | 104 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 36 | x | Yes | 1 – 2 y | 0.216 | -1 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | 1 – 2 y | 0.273 | -0.2 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 40 | | Yes | 1 – 2 y | 0.265 | -0.3 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 73 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | None | 0.253 | -0.4 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | 1 – 2 y | 0.324 | 0.6 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 47 | x | No | None | 0.326 | 0.6 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 74 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.211 | -1 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 49 | x | No | < 1 y | 0.360 | 1.1 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.290 | 0.1 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.304 | 0.3 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | 1 – 2 y | 0.309 | 0.4 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 56 | | No | 1 – 2 y | 0.278 | -0.1 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 57 | | Yes | 1 – 2 y | 0.282 | 0 | 0.025 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 90.3 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 58 | x | No | < 1 y | 0.290 | 0.1 | 0.05 | | | | | | | | | | | | | | No | 107 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 60 | x | Yes | 1 – 2 y | 0.362 | 1.1 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 48.4 % (48.4 %; CV 6.5 %) | SB-EUPT | 3 | |
| 61 | | Yes | 1 – 2 y | 0.279 | -0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 63 | | Yes | > 2 y | 0.244 | -0.6 | 0.01 | 10 | ambient | No | 30 | Other, specify under "Solvent details" | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 64 | x | Yes | > 2 y | 0.281 | 0 | 0.01 | 2 | cold | No | 1 | $H_2O$,ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylam-moniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 65 | | Yes | < 1 y | 0.434 | 2.1 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 93 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |
| 66 | x | No | None | 0.295 | 0.2 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 68 | | No | None | 0.040 | -3.4 | 0.02 | 5 | cold | No | 20 | $H_2O$,ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | - | | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-67 | | No | 1 – 2 y | 0.343 | 0.8 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 93 % (0.5 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-69 | | Yes | 1 – 2 y | 0.310 | 0.4 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, addi-tion of 20 ml of 0.1 % FA solution | No | Freezing out, Centri-fuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 112 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |
| 3rd-71 | | Yes | 1 – 2 y | 0.320 | 0.5 | 0.01 | 2 | ambient | No | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | 1 – 2 y | 0.318 | 0.6 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TPP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 2 | x | No | None | 0.330 | 0.7 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.291 | 0.2 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/$MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 97 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 5 | x | Yes | 1 – 2 y | 0.308 | 0.4 | 0.005 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.281 | 0 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 7 | x | No | 1 – 2 y | 0.219 | -0.9 | 0.01 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 8 | | Yes | 1 – 2 y | 0.275 | -0.1 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, QuEChERS citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |
| 9 | x | No | 1 – 2 y | 0.247 | -0.5 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.309 | 0.4 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.205 | -1.1 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, BAC 12 D6 | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | No | > 2 y | 0.315 | 0.5 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 115.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.32 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.291 | 0.2 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 93 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.4 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.262 | -0.3 | 0.1 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | No | 1 – 2 y | 0.117 | -2.3 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | No | 1 – 2 y | 0.282 | 0 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, addition of QuEChERS citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 25 | | Yes | 1 – 2 y‡ | 0.300 | 0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 100.1 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | 1 – 2 y | 0.296 | 0.2 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation,only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | No | 1 – 2 y | 0.200 | -1.1 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | 1 – 2 y | 0.223 | -0.8 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.075 | -2.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | 1 – 2 y | 0.265 | -0.2 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 35 | | Yes | 1 – 2 y | 0.302 | 0.3 | 0.02 | 5 | ambient | No | 10 | $H_2O$, ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltrimethylammonium chloride | No | 103 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 36 | x | Yes | 1 – 2 y | 0.240 | -0.6 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | 1 – 2 y | 0.298 | 0.3 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 40 | | Yes | 1 – 2 y | 0.285 | 0.1 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 77 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | None | 0.192 | -1.3 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | 1 – 2 y | 0.300 | 0.3 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 47 | x | No | None | 0.297 | 0.3 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 70 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.222 | -0.8 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 49 | x | No | < 1 y | 0.350 | 1 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.292 | 0.2 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.335 | 0.8 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | 1 – 2 y | 0.286 | 0.1 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 56 | | No | 1 – 2 y | 0.278 | 0 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 57 | | Yes | 1 – 2 y | 0.280 | 0 | 0.01 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 89.6 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | No | < 1 y | 0.340 | 0.9 | 0.05 | | | | | | | | | | | | | | No | 113 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 60 | x | Yes | 1 – 2 y | 0.358 | 1.1 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 45.8 % (45.8 %; C.V. =5.5 %) | SB-EUPT | 3 | |
| 61 | | Yes | 1 – 2 y | 0.252 | -0.4 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 63 | | Yes | > 2 y | 0.233 | -0.7 | 0.01 | 10 | ambient | No | 30 | Other, not specified | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 64 | x | Yes | > 2 y | 0.286 | 0.1 | 0.01 | 2 | cold | No | 1 | $H_2O$, ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylammoniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 65 | | Yes | < 1 y | 0.878 | 8.6 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 94 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |
| 66 | x | No | None | 0.277 | 0 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 68 | | No | None | 0.046 | -3.3 | 0.02 | 5 | cold | No | 20 | $H_2O$ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | - | | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-67 | | No | 1 – 2 y | 0.323 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 96 % (0.5 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-69 | | Yes | 1 – 2 y | 0.330 | 0.7 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, addition of 20 ml of 0.1 % FA solution | No | Freezing out, Centrifuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 90 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |
| 3rd-71 | | Yes | 1 – 2 y | 0.300 | 0.3 | 0.01 | 2 | ambient | No | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | > 2 y* | 0.199 | 0.4 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.158 | -0.5 | 0.02 | 10 | ambient | No | 10 | MeOH : $H_2O$ : HOAc = 75:24:1 | No | No | Centrifugation | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | in house method |
| 4 | | Yes | < 1 y# | 0.118 | -1.4 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 70 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.180 | 0 | 0.02 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.168 | -0.2 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | |
| 7 | x | No | None | 0.179 | 0 | 0.02 | 10 | cold | No | 1 | ACN | No | No | Dispersive-SPE, C18/ PSA | | LC-MS | | Std. add to sample portions | | Yes-2 | | | | |
| 8 | | No | > 2 y§ | 0.175 | -0.1 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 9 | x | No | None | 0.138 | -0.9 | 0.01 | 5 | ambient | No | 45 | MeOH,$H_2O$/HCl | No | No | Freezing out, SPE - column (C18) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, chlormequat chloridD4 | No | | SB-EUPT | 2 | |
| 10 | | Yes | > 2 y¶ | 0.298 | 2.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.179 | 0 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | Yes | > 2 y | 0.199 | 0.4 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 122.1 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.205 | 0.6 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.172 | -0.2 | 0.01 | 10 | cold | No | 1 | Other, not specified | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | Yes-3 | 95 % (0.143 mg/kg) | SB-EUPT | 3 | Waters Appl. Note |
| 18 | x | Yes | > 2 y | 0.116 | -1.4 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 107.5 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 21 | | Yes | > 2 y | 0.240 | 1.4 | 0.01 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 22 | | No | > 2 y | 0.137 | -0.9 | 0.02 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.131 | -1.1 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.195 | 0.4 | 0.02 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA) ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 25 | | Yes | > 2 y‡ | 0.153 | -0.6 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS | PS-ML | No | No | 107.6 % (0.1 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ¶ no experience with analytical column; § no experience with matrix ofanimal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 26 | | Yes | > 2 y | 0.146 | -0.7 | 0.005 | 10 | ambient | No | 5 | MeOH, $H_2O$ | | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | DIN EN 15055, 2006-08 |
| 27 | | No | > 2 y | 0.279 | 2.2 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 28 | | No | > 2 y | 0.200 | 0.5 | 0.005 | 5 | ambient | | 30 | MeOH | | | Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 30 | | No | > 2 y | 0.242 | 1.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.197 | 0.4 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.183 | 0.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 35 | | No | None | 0.180 | 0 | 0.01 | 5 | ambient | No | 20 | $H_2O$, MeOH | No | No | None | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 37 | | No | > 2 y | 0.186 | 0.2 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 38 | x | Yes | > 2 y | 0.164 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.170 | -0.2 | 0.02 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 41 | x | No | < 1 y | 0.207 | 0.6 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 43 | | No | > 2 y | 0.229 | 1.1 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.180 | 0 | 0.005 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | §64 LFGB L 00.00-76 |
| 46 | | No | > 2 y | 0.197 | 0.4 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, method QuPPe LC 3 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | < 1 y | 0.216 | 0.8 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 76 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.178 | 0 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM, as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | 1 – 2 y[§] | 0.170 | -0.2 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.216 | 0.8 | 0.02 | 10 | just thawed | No | 20 | Other, Not specified | No | No | Dispersive-SPE, instead of ODS Z-Sep+ was used | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 51 | x | Yes | > 2 y | 0.106 | -1.6 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; [#] Experience referring to commodity "milk"; [¶] no experience with analytical column; [§] no experience with matrix ofanimal origin; [‡] No experience with milk; [$] Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 52 | x | No | > 2 y | 0.172 | -0.2 | 0.01 | 20 | ambient | No | 3 | MeOH | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | DIN EN 15055 |
| 53 | x | No | < 1 y | 0.144 | -0.8 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 54 | | No | > 2 y | 0.170 | -0.2 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Clormequat-D4 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.170 | -0.2 | 0.02 | 1 | ambient | No | 1 | DCMe, HOAc | Yes, once, add hydroxide ammonium just before clean up | No | Centrifugation, SPE-column (ion exchange), double Centrifugation, Filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, chlormequat D4 | Yes-4 | | SB-EUPT | 2 | in house method |
| 56 | | Yes | > 2 y | 0.179 | 0 | 0.004 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, HPLC, Quatrro Premier, ESI+ | None | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 4 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 58 | x | No | None | 0.160 | -0.4 | 0.01 | 10 | cold | No | 20 | Other, specify under "Solvent details" | No | No | Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | 99 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 59 | | Yes | 1 – 2 y | 0.183 | 0.1 | 0.02 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, Isoproturon-D6 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 61 | | Yes | > 2 y | 0.211 | 0.7 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, CCC-D4 | No | | SB-EUPT | 2 | Methanol extraction |
| 62 | | Yes | > 2 y | 0.150 | -0.7 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 39 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.247 | 1.5 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.177 | 0 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 66 | x | Yes | > 2 y | 0.090 | -2 | 0.02 | 10 | cold | No | 2 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 3rd-33 | | Yes | 1 – 2 y | 0.160 | -0.4 | 0.02 | 2 | ambient | No | 5 | Other, specify under "Solvent details" | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 77.7 % (0.02 mg/kg) | SB-EUPT | 1 | in house method |
| 3rd-71 | | Yes | > 2 y | 0.340 | 3.6 | 0.01 | 3 | ambient | Yes, Complete dryness | 10 | Other, specify under "Solvent details" | No | | | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Restek Article |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ¶ no experience with analytical column; § no experience with matrix ofanimal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | 1 – 2 y | 0.320 | 0.8 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TPP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 2 | x | No | None | 0.276 | 0.1 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.305 | 0.6 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/$MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 98 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 5 | x | Yes | 1 – 2 y | 0.285 | 0.3 | 0.005 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.215 | -0.8 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 7 | x | No | 1 – 2 y | 0.284 | 0.2 | 0.01 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 8 | | Yes | 1 – 2 y | 0.254 | -0.2 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |
| 9 | x | No | 1 – 2 y | 0.239 | -0.4 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.289 | 0.3 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.217 | -0.8 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | No | > 2 y | 0.339 | 1.1 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 109.2 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.301 | 0.5 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.301 | 0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 96 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.3 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.278 | 0.1 | 0.1 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | No | 1 – 2 y | 0.118 | -2.2 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | No | 1 – 2 y | 0.321 | 0.8 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 25 | | Yes | 1 – 2 y‡ | 0.289 | 0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 100.4 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 26 | | Yes | 1 – 2 y | 0.310 | 0.6 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation,only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | No | 1 – 2 y | 0.234 | -0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | 1 – 2 y | 0.235 | -0.5 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.097 | -2.6 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | 1 – 2 y | 0.161 | -1.6 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 35 | | Yes | 1 – 2 y | 0.255 | -0.2 | 0.02 | 5 | ambient | No | 10 | $H_2O$ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltrimethylammonium chloride | No | 103 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 36 | x | Yes | 1 – 2 y | 0.276 | 0.1 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | 1 – 2 y | 0.272 | 0.1 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | Yes-1 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 40 | | Yes | 1 – 2 y | 0.249 | -0.3 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 90 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | None | 0.129 | -2.1 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | 1 – 2 y | 0.299 | 0.5 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 47 | x | No | None | 0.253 | -0.2 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 74 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.190 | -1.2 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 49 | x | No | < 1 y | 0.250 | -0.3 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | - | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.343 | 1.1 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.310 | 0.6 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | 1 – 2 y | 0.189 | -1.2 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 56 | | No | 1 – 2 y | 0.302 | 0.5 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 57 | | Yes | 1 – 2 y | 0.264 | -0.1 | 0.01 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 91.7 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | No | < 1 y | 0.240 | -0.4 | 0.05 | | | | | | | | | | | | | | No | 114 % (0.05 mg/kg) | SB-EUPT | 1 | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 60 | x | Yes | 1 – 2 y | 0.349 | 1.2 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 45.6 % (45.6 %; C.V% = 4.5 %) | SB-EUPT | 3 | |
| 61 | | Yes | 1 – 2 y | 0.267 | 0.0 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 63 | | Yes | > 2 y | 0.227 | -0.6 | 0.01 | 10 | ambient | No | 30 | Other, not specified | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 64 | x | Yes | > 2 y | 0.239 | -0.4 | 0.01 | 2 | cold | No | 1 | $H_2O$, ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylam-moniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 65 | | Yes | < 1 y | 0.313 | 0.7 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 96 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |
| 68 | | No | None | 0.306 | 0.6 | 0.02 | 5 | cold | No | 20 | $H_2O$, ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-69 | | Yes | 1 – 2 y | 0.400 | 2.0 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, addi-tion of 20 ml of 0.1 % FA solution | No | Freezing out, Centri-fuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 62 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |
| 3rd-71 | | Yes | 1 – 2 y | 0.270 | 0.0 | 0.01 | 2 | ambient | No | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | > 2 y* | 0.094 | -1.8 | 0.01 | 10 | cold | | 30 | ACN | No | No | | | LC-MS/MS QQQ | | MM-ML | Yes, nicarbazim | Yes-5 | | | | QuEChERS for acidic pesticides (EURL-SRM method) |
| 2 | x | No | > 2 y | 0.189 | 0.5 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.187 | 0.4 | 0.005 | 10 | cold | No | 15 | ACN | Yes, once, citrat buffer, pH 5 | No | Centrifugation, Freezing out, Filtration through syringe filter | | LC-MS/MS QQQ, ESI negativ | | MM-ML | Yes, nicarbazin | No | 101 % (0.2 mg/kg) | SB-EUPT | 3 | Analysis of Phenoxyalkanoic Acids in Milk using QuECgERS method an LC-MS/MS |
| 4 | | Yes | < 1 y# | 0.175 | 0.1 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-Orbitrap | None | MM-ML | Yes, Mecoprop D3 | No | 104 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.187 | 0.4 | 0.01 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.116 | -1.3 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 8 | | Yes | > 2 y | 0.154 | -0.4 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to extract aliquots | No | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | None | 0.188 | 0.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | > 2 y‡ | <0.01 | -3.8 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | > 2 y | 0.154 | -0.4 | 0.01 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | Yes | > 2 y | 0.27 | 2.4 | 0.01 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 125.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | > 2 y | 0.203 | 0.8 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.19 | 0.5 | 0.02 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 97 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 17 | | No | None | 0.11 | -1.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | Yes-5 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.0803 | -2.1 | 0.01 | 10 | just thawed | No | 1 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ, Zorbax XDB-C18 | Std. add to extract aliquots | No | Yes-1 | 91.5 % (0.01 mg/kg) | SB-EUPT | 4 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 21 | | Yes | > 2 y | 0.16 | -0.2 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | > 2 y | 0.175 | 0.1 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TMA | Yes-3 | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 23 | | No | 1 – 2 y | 0.157 | -0.3 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.194 | 0.6 | 0.01 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 25 | | Yes | > 2 y‡ | 0.194 | 0.6 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 107.1 % (0.04 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.203 | 0.8 | 0.01 | 25 | ambient | Yes, alkine with NaOH | > 60 | Acetone, CyHn, EtOAc, first hydrolysis | Yes, once | Yes, with tetrabutyl-ammonium-hydroxid/ isomethane | GPC, Gel-Permeation Chr/phy, acid/base distribution | | | GC-MSD, via second m/z | PS-SL | Yes, d3-Mecoprop, no calculation | No | | SB-EUPT | 1 | alkine hydrolysis extraction GPC, acid/base distribution, methykation,GC-MSD detection |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 27 | | Yes | > 2 y | 0.142 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 30 | | Yes | > 2 y | 0.218 | 1.1 | 0.01 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.15 | -0.5 | 0.01 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.133 | -0.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | > 2 y | 0.137 | -0.8 | | 5 | just thawed | | 10 | EtOAc | Yes, once, HOAc | | Dessication with $Na_2SO_4$, Z-Sep/C18 | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | |
| 37 | | No | > 2 y | 0.217 | 1.1 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 38 | x | Yes | 1 – 2 y | 0.131 | -0.9 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.208 | 0.9 | 0.01 | 10 | | | 15 | ACN | No | | Freezing out, 2h | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazine | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 41 | x | No | > 2 y | 0.106 | -1.5 | 0.01 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 43 | | Yes | > 2 y | 0.158 | -0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.173 | 0.1 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 46 | | No | > 2 y | 0.0965 | -1.7 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extraction solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, 'standard' C18 R.P. chromatography | None | MM-SL | No | No | | | | Extraction with ACN + 1 % FA. No partitioning step |
| 47 | x | No | None | 0.184 | 0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 104 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | > 2 y | 0.152 | -0.4 | 0.01 | 5 | cold | No | 10 | ACN | No | No | None | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | | | |
| 49 | x | No | > 2 y | 0.2 | 0.7 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 50 | x | Yes | > 2 y | 0.266 | 2.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | No | | SB-EUPT | 1 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 51 | x | Yes | > 2 y | 0.202 | 0.8 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Dispersive-SPE (ODS/ $MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-2 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 52 | x | No | > 2 y | 0.133 | -0.9 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.225 | 1.3 | 0.01 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | No | > 2 y | 0.141 | -0.7 | 0.01 | 5 | ambient | No | 20 | EtOAc | Yes, once | No | z-sep+ | | | None | MM-SL | Yes, Pirimicarb-D6 | No | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |
| 55 | x | Yes | > 2 y | 0.132 | -0.9 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 56 | | Yes | > 2 y | 0.137 | -0.8 | 0.002 | 10 | ambient | No | 15 | ACN | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI+ | None | MM-ML | Yes, TPP | No | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 58 | x | Yes | > 2 y | 0.062 | -2.5 | 0.02 | | | | | | | | | | | | | | No | 41 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 59 | | Yes | 1 – 2 y | 0.012 | -3.7 | 0.01 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, terbuthylazine D5 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 60 | x | Yes | > 2 y | 0.212 | 1 | 0.02 | 10 | deep frozen | No, None | 10 | ACN | No, None | No, None | Freezing out | | LC-MS/MS QQQ, None | None | MM-SL | Yes, nicarbazin | No | 92.5 % (92 %; C.V% 2.8 %) | SB-EUPT | 3 | |
| 61 | | Yes | > 2 y | 0.175 | 0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 62 | | Yes | > 2 y | 0.19 | 0.5 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | 104.4 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.191 | 0.5 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, nicarbazin | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.154 | -0.4 | 0.01 | 5 | cold | No | 1 | ACN | Yes, once, 1 % HOAc in extraction solvent | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | |
| 66 | x | Yes | > 2 y | 0.332 | 3.8 | 0.02 | 10 | cold | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 3rd-33 | | No | 1 – 2 y | 0.215 | 1.1 | 0.01 | 5 | ambient | No | 5 | QuPPe solvent (MeOH/1 % FA) | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 93 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-71 | | Yes | < 1 y | 0.29 | 2.8 | 0.01 | 3 | ambient | Yes, Complete dryness | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazim | Yes-1 | | SB-EUPT | 2 | Journal of Environmental Science and Health Part B |
| 3rd-72 | | No | 1 – 2 y | 0.202 | 0.8 | 0.01 | 10 | cold | No | 1 | ACN | Yes, once | No | Dispersive-SPE | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Maleic hydrazide (Assigned Value = 0.342 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | | FN | -3.4 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | < 1 y | 0.407 | 0.8 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 4 | | No | < 1 y# | 0.327 | -0.2 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 102 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.369 | 0.3 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 8 | | No | None | 0.342 | 0.0 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 9 | x | No | None | 0.374 | 0.4 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | No | None | 0.343 | 0.0 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.381 | 0.5 | 0.05 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | > 2 y | 0.316 | -0.3 | 0.05 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 111.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | < 1 y | 0.251 | -1.1 | 0.05 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.192 | -1.8 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 110.5 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 21 | | Yes | > 2 y | 0.400 | 0.7 | 0.05 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 22 | | No | None | 0.918 | 6.7 | 0.1 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.606 | 3.1 | 0.1 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.274 | -0.8 | 0.05 | 10 | ambient | No | 1 | MeOH | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 31 | x | No | < 1 y | 0.337 | -0.1 | 0.1 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.250 | -1.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 37 | | No | > 2 y | 0.338 | 0.0 | 0.1 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 38 | x | No | < 1 y | 0.309 | -0.4 | 0.1 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Maleic hydrazide (Assigned Value = 0.342 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 39 | x | No | < 1 y | 0.261 | -0.9 | 0.05 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 46 | | No | None | 0.361 | 0.2 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, QuPPe LC method 2 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | None | 0.346 | 0.0 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 102 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | None | 0.379 | 0.4 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | | FN | -3.4 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 52 | x | No | None | 0.416 | 0.9 | 0.02 | 10 | ambient | No | 15 | MeOH | Yes, once | No | Liquid-liquid partitioning | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.273 | -0.8 | 0.05 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D2 | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 54 | | No | > 2 y | 0.346 | 0.0 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Maleic hydrazide-D2 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.344 | 0.0 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, maleic hydrazide D2 | Yes-4 | | SB-EUPT | 1 | in house method |
| 61 | | Yes | > 2 y | 0.363 | 0.2 | 0.1 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | MethaNol extraction |
| 63 | | Yes | 1 – 2 y | 0.229 | -1.3 | 0.05 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-33 | | Yes | 1 – 2 y | 0.551 | 2.4 | 0.05 | 5 | ambient | No | 5 | QuPPe solvent (MeOH/1 % FA) | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | > 2 y* | 0.34 | 0.1 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.302 | -0.4 | 0.02 | 10 | ambient | No | 10 | MeOH : $H_2O$ : HOAc = 75:24:1 | No | No | Centrifugation | | LC-MS/MS QQQ | | MM-ML | Yes, Chlormequat C13 | No | | SB-EUPT | 1 | in house method |
| 4 | | Yes | < 1 y# | 0.306 | -0.3 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 108 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.323 | -0.1 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Mepiquat D3 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | > 2 y | 0.301 | -0.4 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | |
| 7 | x | No | None | 0.36 | 0.3 | 0.02 | 10 | cold | No | 1 | ACN | No | No | Dispersive-SPE, C18/ PSA | | LC-MS | | Std. add to sample portions | | Yes-2 | | | | |
| 8 | | No | > 2 y§ | 0.335 | 0.0 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 9 | x | No | None | 0.248 | -1.0 | 0.01 | 5 | ambient | No | 45 | MeOH, $H_2O$/HCl | No | No | Freezing out, SPE - column (C18) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | |
| 10 | | No | None | 0.472 | 1.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.357 | 0.3 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | Yes | > 2 y | 0.37 | 0.4 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 118.4 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.361 | 0.3 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.349 | 0.2 | 0.01 | 10 | cold | No | 1 | Other, not specified | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | Yes-3 | 90 % (0.257 mg/kg) | SB-EUPT | 3 | Waters Appl. Note |
| 18 | x | Yes | > 2 y | 0.196 | -1.6 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 106.2 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 21 | | Yes | > 2 y | 0.43 | 1.2 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 22 | | No | > 2 y | 0.255 | -0.9 | 0.01 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.290 | -0.5 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.368 | 0.4 | 0.02 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA) ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 25 | | Yes | > 2 y‡ | 0.264 | -0.8 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS | PS-ML | No | No | 97.0 % (0.1 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.292 | -0.5 | 0.005 | 10 | ambient | No | 5 | MeOH, $H_2O$ | | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | DIN EN 15055, 2006-08 |
| 27 | | No | > 2 y | 0.708 | 4.5 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 28 | | No | > 2 y | 0.450 | 1.4 | 0.005 | 5 | ambient | | 30 | MeOH | | | Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 30 | | No | > 2 y | 0.429 | 1.2 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.375 | 0.5 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, chlormequat | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 35 | | No | None | 0.362 | 0.3 | 0.01 | 5 | ambient | No | 20 | $H_2O$, MeOH | No | No | None | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 37 | | No | > 2 y | 0.301 | -0.4 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 38 | x | Yes | > 2 y | 0.311 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.388 | 0.7 | 0.02 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 41 | x | No | < 1 y | 0.467 | 1.6 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 43 | | No | > 2 y | 0.466 | 1.6 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.330 | 0.0 | 0.005 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | §64 LFGB L 00.00-76 |
| 46 | | No | > 2 y | 0.342 | 0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, QuPPe LC method 3 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | < 1 y | 0.359 | 0.3 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 81 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.320 | -0.2 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | 1 – 2 y | 0.280 | -0.6 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.358 | 0.3 | 0.02 | 10 | just thawed | No | 20 | Other, not specified | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 51 | x | Yes | > 2 y | 0.219 | -1.4 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; § Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 52 | x | No | > 2 y | 0.324 | -0.1 | 0.01 | 20 | ambient | No | 3 | MeOH | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | DIN EN 15055 |
| 53 | x | No | < 1 y | 0.294 | -0.5 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D3 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 54 | | No | > 2 y | 0.326 | -0.1 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Mepiquat-D3 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.307 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, chlormequat D4 | Yes-2 | | SB-EUPT | 2 | in house method |
| 56 | | Yes | > 2 y | 0.343 | 0.1 | 0.004 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, HPLC, Quatrro Premier, ESI+ | None | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 4 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 58 | x | No | None | 0.300 | -0.4 | 0.01 | | | | | | | | | | | | | | No | 65 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 59 | | Yes | 1 – 2 y | 0.273 | -0.7 | 0.02 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, Isoproturon-D6 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 61 | | Yes | > 2 y | 0.400 | 0.8 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, CCC-D4 | No | | SB-EUPT | 2 | Methanol extraction |
| 62 | | Yes | > 2 y | 0.300 | -0.4 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 52.5 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.334 | 0 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, IL-target pesticide | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.377 | 0.5 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 66 | x | Yes | > 2 y | 0.102 | -2.8 | 0.02 | 10 | cold | No | 2 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 3rd-33 | | Yes | 1 – 2 y | 0.300 | -0.4 | 0.02 | 2 | ambient | No | 5 | Other, not specified | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 79.5 % (0.02 mg/kg) | SB-EUPT | 1 | in house method |
| 3rd-71 | | Yes | > 2 y | 0.360 | 0.3 | 0.01 | 3 | ambient | Yes, Complete dryness | 10 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Restek Article |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Chlorate (Assigned Value = 0.185 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | < 1 y* | 0.175 | -0.2 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | None | 0.202 | 0.4 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 4 | | Yes | < 1 y# | 0.178 | -0.2 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 103 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | No | < 1 y | 0.180 | -0.1 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, provided by organizer | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | < 1 y | 0.143 | -0.9 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-1 | 95 % | QC | >5 | |
| 7 | x | No | | FN | -3.6 | 0.1 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | | | | | |
| 9 | x | No | None | 0.181 | -0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | Yes | < 1 y | 0.195 | 0.2 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | < 1 y | 0.210 | 0.5 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | < 1 y | 0.276 | 2.0 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | Yes, IL-target pesticide | Yes-1 | 59.6 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | 1 – 2 y | 0.185 | 0.0 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.180 | -0.1 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 23 | | No | 1 – 2 y | 0.159 | -0.6 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 26 | | Yes | < 1 y | 0.162 | -0.5 | 0.01 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, calculated with delivered ISTD | No | | SB-EUPT | 1 | |
| 30 | | No | None | 0.207 | 0.5 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | No | < 1 y | 0.209 | 0.5 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Freezing out | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 35 | | No | None | 0.187 | 0.0 | 0.02 | 10 | ambient | No | 10 | $H_2O$, QuPPe solvent (MeOH/1 % FA), ACN | No | No | Dispersive-SPE (ODS), 1 min shaking | | LC-MS/MS QQQ, AJS ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 98 % (0.2 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 37 | | No | < 1 y | 0.135 | -1.1 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 44 | | Yes | < 1 y | 0.16 | -0.5 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Chlorate (Assigned Value = 0.185 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 47 | x | No | None | 0.172 | -0.3 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-SL | No | No | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | None | 0.203 | 0.4 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | < 1 y | 0.186 | 0.0 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transi-tions | None | PS-ML | Yes, IL-target pesticide | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.152 | -0.7 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ion chromatog-raphy | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, 18 O 4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 56 | | No | None | 0.235 | 1.1 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addi-tion of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI- | None | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 61 | | Yes | 1 – 2 y | 0.236 | 1.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, Perchlorate 1804 | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 62 | | No | | FN | -3.6 | 0.05 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | | | | | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | 1 – 2 y | 0.252 | 1.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample por-tions | No | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.178 | -0.2 | 0.01 | 2 | cold | No | 1 | ACN, $H_2O$ | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | extraction with ACN/0.1M FA (20/80 v/v) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Cyromazine (Assigned Value = 0.230 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | > 2 y* | 0.229 | 0.0 | 0.01 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | 1 – 2 y | 0.361 | 2.3 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 4 | | Yes | < 1 y# | 0.224 | -0.1 | 0.01 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 101 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.252 | 0.4 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Cyromazine D4 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | > 2 y | 0.151 | -1.4 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 8 | | Yes | 1 – 2 y | 0.172 | -1.0 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 10 | | Yes | > 2 y‡ | 0.109 | -2.1 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.278 | 0.8 | 0.01 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | > 2 y | 0.254 | 0.4 | 0.01 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 93.5 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.280 | 0.9 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.145 | -1.5 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 101.4 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 21 | | Yes | > 2 y | 0.250 | 0.4 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE (PSA/$MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | No | 1 – 2 y | 0.220 | -0.2 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | No | < 1 y | 0.307 | 1.3 | 0.01 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA), ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 27 | | Yes | > 2 y | 0.179 | -0.9 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | Std. add to sample portions | Yes, TDCP | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 28 | | No | > 2 y | 0.097 | -2.3 | 0.01 | 5 | ambient | | 10 | ACN | | | Filtration | | | GC-MS/MS (QQQ) | MM-ML | No | No | | | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 30 | | No | > 2 y | 0.304 | 1.3 | 0.01 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | No | 1 – 2 y | 0.355 | 2.2 | 0.01 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, chlormequat | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.287 | 1.0 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 34 | x | No | > 2 y | 0.195 | -0.6 | | 5 | just thawed | | 30 | EtOAc | No | | Filtration + Std. add to sample portions, Filtration, PSA/C18 | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | Std. add to sample portions | | Yes-2 | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Cyromazine (Assigned Value = 0.230 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 38 | x | No | 1 – 2 y | 0.268 | 0.7 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 41 | x | No | < 1 y | 0.193 | -0.6 | 0.01 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, mepiquat | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 44 | | Yes | > 2 y | 0.121 | -1.9 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 46 | | No | > 2 y | 0.224 | -0.1 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extraction solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, QuPPe LC method 3 | None | MM-SL | No | Yes-3 | | | | Extraction with ACN+1 % FA. No partitioning step |
| 47 | x | No | < 1 y | 0.214 | -0.3 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 96 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.196 | -0.6 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | 1 – 2 y | 0.32 | 1.6 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.252 | 0.4 | 0.01 | 10 | just thawed | No | 20 | Other, not specified | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 4 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 51 | x | Yes | 1 – 2 y | 0.138 | -1.6 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 52 | x | No | > 2 y | 0.285 | 1.0 | 0.01 | 10 | ambient | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 55 | x | Yes | > 2 y | 0.221 | -0.1 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |
| 56 | | No | 1 – 2 y | 0.305 | 1.3 | 0.01 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 58 | x | No | None | 0.17 | -1.0 | 0.02 | | | | | | | | | | | | | | No | 92 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 61 | | Yes | > 2 y | 0.248 | 0.3 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 62 | | No | > 2 y | 0.19 | -0.7 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 60.7 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.197 | -0.6 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, trifenylfosfaat | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.253 | 0.4 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 66 | x | No | < 1 y | 0.275 | 0.8 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 3rd-33 | | Yes | > 2 y | 0.186 | -0.8 | 0.01 | 30 | ambient | No | 5 | EtOAc | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 99.5 % (0.01 mg/kg) | SB-EUPT | 1 | in house method |
| 3rd-67 | | Yes | > 2 y | 0.163 | -1.2 | 0.01 | 5 | cold | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | Yes, PCB 31 | Yes-1 | 36 % (0.1 mg/kg) | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Melamine (Assigned Value = 0.365 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | < 1 y | 0.324 | -0.5 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | No | < 1 y# | 0.351 | -0.2 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 92 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | No | < 1 y | 0.349 | -0.2 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Melamine tramine 15N3 | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 8 | | No | None‡ | 0.347 | -0.2 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA), ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 10 | | Yes | > 2 y‡ | 0.394 | 0.3 | 0.05 | 1 | ambient | No | 5 | ACN | No | No | | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 4 | |
| 11 | | No | None | 0.364 | 0.0 | | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.340 | -0.3 | 0.05 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 20 | x | Yes | > 2 y | 0.396 | 0.3 | 0.15 | 5 | deep frozen | No | 15 | Diethylamine /$H_2O$/ACN | No | Yes, with BSTFA and 1 % TMCS | Filtration | | GC-MS/MS QQQ | GC-MS/MS (QQQ) | MM-ML | Yes, 2.6 Diamino-4-chloropyrimidine | Yes-1 | | SB-EUPT | 5 | GC-MS Screen for the presence of melamine and related analogs - US FDA |
| 21 | | Yes | < 1 y | 0.500 | 1.5 | 0.05 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 27 | | No | > 2 y | FN | -3.5 | 1 | 1 | cold | No | 1 | ACN:$H_2O$ = 50:50 | | | SPE, MycoSep (RomerLabs) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | - | 1 | |
| 44 | | Yes | > 2 y | 0.396 | 0.3 | 0.25 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | in house method |
| 46 | | No | > 2 y | 0.399 | 0.4 | 0.05 | 5 | ambient | No | 5 | ACN | No | No | None | | LC-MS/MS QQQ, HILIC chromatograpy | None | PS-ML | Yes, labbeld analogue | Yes-3 | | | | Extraction with ACN |
| 47 | x | No | < 1 y | 0.375 | 0.1 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-ML | Yes, IL-target pesticide | Yes-4 | 95 % (0.050 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.362 | 0.0 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 51 | x | No | None | 0.222 | -1.6 | 0.05 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 59 | | Yes | > 2 y | 0.360 | -0.1 | 0.1 | 2 | deep frozen | No | 30 | ACN, $H_2O$ | No | Yes, with MSTF at 60 °C | Centrifugation, Filtration | | | GC-MS/MS (QQQ) | MM-SL | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | LNR LABERCA/08MEL-AI.7 |
| 61 | | No | None | 0.305 | -0.7 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 63 | | No | None | 0.394 | 0.3 | 0.05 | 10 | ambient | No | 30 | ACN | | No | Quechers citrate buffer mix, Quechers citrate buffer mix | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | - | 3 | Chlorothalonil: QuEChERS-based mth by EURL-SRM |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: # Experience referring to commodity "milk"; ‡ Experience only with partcipation in QuPPe Interlaboratory Study ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Melamine (Assigned Value = 0.365 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 64 | x | Yes | < 1 y | 0.375 | 0.1 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-33 | | Yes | > 2 y | 0.362 | 0.0 | 0.05 | 1 | ambient | No | 5 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 106 % (0.05 mg/kg) | SB-EUPT | 1 | in house method |
| 3rd-67 | | Yes | > 2 y | 0.411 | 0.5 | 0.1 | 3 | cold | No | 10 | Other, not specified | No | No | None | | | None | MM-ML | No | No | 107 % (0.5 mg/kg) | | 4 | Technical Specification ISO/TS 15495 IDF/RM230 |
| 3rd-69 | | Yes | > 2 y | 0.280 | -0.9 | 0.1 | 10 | cold | No | 30 | ACN, Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, Added 0.5 ml of HCl and 0.5 ml NaOH to adjust pH to 7 | No | Filtration, 0.45um PTFE filter | | | LC-MS/MS QQQ | PS-ML | Yes, 15N3 | Yes-1 | | SB-EUPT | 2 | in house method |
| 3rd-72 | | No | 1 – 2 y | 0.292 | -0.8 | 0.5 | 5 | cold | No | 10 | ACN, other (not specified) | No | No | None | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: ♯ Experience referring to commodity "milk"; ♯♯ Experience only with partcipation in QuPPe Interlaboratory Study ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Perchlorate (Assigned Value = 0.180 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | 1 – 2 y* | 0.265 | 1.9 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | None | 0.196 | 0.3 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 4 | | Yes | < 1 y# | 0.181 | 0.0 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 102 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.151 | -0.7 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, provided by organizer | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | 1 – 2 y | 0.127 | -1.2 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 7 | x | No | None | 0.132 | -1.1 | 0.02 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 9 | x | No | None | 0.184 | 0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | Yes | < 1 y | 0.250 | 1.5 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | < 1 y | 0.204 | 0.5 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | < 1 y | 0.171 | -0.2 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | Yes, IL-target pesticide | No | 92.5 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | 1 – 2 y | 0.210 | 0.7 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.170 | -0.2 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 23 | | No | 1 – 2 y | 0.126 | -1.2 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 26 | | Yes | < 1 y | 0.184 | 0.1 | 0.01 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, calculated with delivered ISTD | No | | SB-EUPT | 1 | |
| 30 | | No | < 1 y | 0.199 | 0.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | No | < 1 y | 0.217 | 0.8 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Freezing out | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.050 | -2.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 35 | | No | None | 0.193 | 0.3 | 0.02 | 10 | ambient | No | 10 | $H_2O$, QuPPe solvent (MeOH/1 % FA), ACN | No | No | Dispersive-SPE (ODS), 1 min shaking | | LC-MS/MS QQQ, AJS ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 104 % (0.2 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 36 | x | No | None | 0.076 | -2.3 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 37 | | No | < 1 y | 0.158 | -0.5 | 0.01 | 10 | ambient | No | 20 | MeOH, H2O | No | No | Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Perchlorate (Assigned Value = 0.180 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 44 | | Yes | < 1 y | 0.180 | 0.0 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | None | 0.560 | 8.4 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-SL | No | No | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | None | 0.171 | -0.2 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | < 1 y | 0.198 | 0.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transi-tions | None | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.156 | -0.5 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ion chromatog-raphy | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, 18 O 4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 56 | | No | None | 0.226 | 1.0 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addi-tion of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI- | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 61 | | Yes | 1 – 2 y | 0.208 | 0.6 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, Perchlorate 1804 | No | | SB-EUPT | 2 | Methanol extraction |
| 62 | | Yes | < 1 y | 0.154 | -0.6 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | 100 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | 1 – 2 y | 0.197 | 0.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample por-tions | Yes, IL-target pesticide | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.177 | -0.1 | 0.01 | 2 | cold | No | 1 | ACN, $H_2O$ | | No | Oasis-Wax | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | extraction with ACN/0.1M FA (20/80 v/v) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### 4-OH-chlorothalonil (Assigned Value for information only = 0.100 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5 | x | No | < 1 y | 0.0916 | -0.3 | 0.01 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | No | None | 0.085 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 21 | | Yes | > 2 y | 0.103 | 0.1 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 47 | x | No | None | 0.111 | 0.4 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 86 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 59 | | Yes | 1 – 2 y | 0.119 | 0.8 | 0.01 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Centrifugation | | | LC-MS/MS QQQ | MM-ML | Yes, nicarbazin | Yes-5 | | SB-EUPT | 1 | Chlorothalonil: QuEChERS-based mth by EURL-SRM |
| 61 | | No | None | 0.078 | -0.9 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 63 | | No | None | 0.109 | 0.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |

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

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Trimesium (Assigned Value for information only = 0.370 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | 1 – 2 y* | 0.569 | 2.2 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | No | < 1 y# | 0.304 | -0.7 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 106 % (0.2 mg/kg) | SB-EUPT | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | No | < 1 y | 0.914 | 5.9 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Trimethylsulfonium D9 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.378 | 0.1 | 0.05 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | No | None | 0.320 | -0.5 | 0.05 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 21 | | Yes | < 1 y | 0.870 | 5.4 | 0.05 | 5 | deep frozen | No | | MeOH, $H_2O$ | No | No | None | | | | MM-ML | No | No | | | | |
| 26 | | Yes | | FN | -3.5 | 0.05 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-SL | No | | | | | |
| 38 | x | No | None | 0.350 | -0.2 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent | No | No | Centrifugation, | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin |
| 47 | x | No | < 1 y | 0.387 | 0.2 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 104 % (0.050 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.387 | 0.2 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, Melamin 15N3 | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 51 | x | No | None | 0.210 | -1.7 | 0.05 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.181 | -0.1 | 0.05 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D9 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | No | < 1 y | 0.480 | 1.2 | 0.1 | 10 | deep frozen | No | 1 | MeOH | No | No | | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 64 | x | Yes | < 1 y | 0.354 | -0.2 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data

## Appendix 7 Methods used by the participating laboratories (ordered by z-scores) .SORY ANALYTES

### 2,4-D (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 18 | x | Yes | > 2 y | 0.0366 | -2.3 | 0.01 | 10 | just thawed | No | 1 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ, Zorbax XDB-C18 | Std. add to extract aliquots | No | Yes-1 | 88.2 % (0.01 mg/kg) | SB-EUPT | 4 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.066 | -1.0 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | Yes | > 2 y | 0.065 | -1.0 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | Yes | 1 – 2 y | 0.068 | -0.9 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 6 | | Yes | > 2 y | 0.070 | -0.8 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | |
| 3rd-71 | | Yes | > 2 y | 0.070 | -0.8 | 0.01 | 5 | ambient | Yes, Evaporate to 1 mL | 20 | Other, no specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | Plant Protection Division Residue Analytical Method No. 68 |
| 3rd-72 | | Yes | 1 – 2 y | 0.070 | -0.8 | 0.01 | 10 | cold | No | 1 | ACN | Yes, once | No | Dispersive-SPE | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | Yes | > 2 y | 0.071 | -0.8 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | Yes | > 2 y | 0.071 | -0.8 | 0.02 | 15 | cold | No | 3 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Filtration | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 23.7 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS - Acetate buffered (AOAC Official Method 2007.01) |
| 10 | | Yes | < 1 y | 0.072 | -0.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 64 | x | Yes | > 2 y | 0.0726 | -0.7 | 0.01 | 5 | cold | No | 1 | ACN | Yes, once, 1 % HOAc in extraction solvent | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS based |
| 56 | | Yes | > 2 y | 0.0744 | -0.6 | 0.002 | 10 | ambient | No | 15 | ACN | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI- | None | MM-ML | Yes, nicarbazim | No | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 38 | x | Yes | 1 – 2 y | 0.0746 | -0.6 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-67 | | Yes | > 2 y | 0.077 | -0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 100 % (0.1 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 53 | x | No | < 1 y | 0.0778 | -0.4 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | > 2 y | 0.078 | -0.4 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.0781 | -0.4 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.079 | -0.4 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 55 | x | Yes | > 2 y | 0.080 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) Other, specify under "Solvent details" | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |
| 43 | | Yes | > 2 y | 0.0803 | -0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662)) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 60 | x | Yes | > 2 y | 0.081 | -0.3 | 0.02 | 10 | deep frozen | No, None | 10 | ACN | No, None | No, None | Freezing out | | LC-MS/MS QQQ, None | None | MM-SL | Yes, nicarbazin | No | 85.3 % (82 %; C.V% 3.5 %) | SB-EUPT | 3 | |
| 48 | x | Yes | > 2 y | 0.082 | -0.3 | 0.02 | 5 | cold | No | 10 | ACN | No | No | None | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | | | |
| 16 | | Yes | < 1 y | 0.0821 | -0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 90 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.083 | -0.2 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 28 | | Yes | > 2 y | 0.083 | -0.2 | 0.01 | 5 | ambient | | 10 | ACN | | | Filtration | | | LC-MS/MS QQQ | MM-SL | No | No | | | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.084 | -0.2 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TMA | Yes-2 | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 1 | | No | > 2 y* | 0.087 | 0.0 | 0.02 | 10 | cold | | 30 | ACN | No | No | | | LC-MS/MS QQQ | | MM-ML | Yes, nicarbazim | Yes-5 | | | | QuEChERS for acidic pesticides (EURL-SRM method) |
| 4 | | Yes | < 1 y# | 0.0875 | 0.0 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-Orbitrap | None | MM-ML | Yes, Mecoprop D3 | No | 98 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.088 | 0.0 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 49 | x | No | > 2 y | 0.088 | 0.0 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 54 | | Yes | > 2 y | 0.088 | 0.0 | 0.01 | 5 | ambient | No | 20 | EtOAc | Yes, once | No | z-sep+ | | | None | MM-SL | Yes, Pirimicarb-D6 | Yes-1 | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |
| 52 | x | No | > 2 y | 0.0881 | 0.0 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 61 | | Yes | > 2 y | 0.0899 | 0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 26 | | Yes | > 2 y | 0.091 | 0.2 | 0.01 | 25 | ambient | Yes, alkine with NaOH | > 60 | Acetone, CyHn, EtOAc, first hydrolysis | Yes, once | Yes, with tetrabutyl-ammonium-hydroxid/ iosomethane | GPC, Gel-Permeation Chr/phy, acid/base distribution | | | GC-MSD, via second m/z | PS-SL | Yes, d3-Mecoprop, no calculation | No | | SB-EUPT | 1 | alkine hydrolysis extraction GPC, acid/base distribution, methykation,GC-MSD detection |
| 5 | x | Yes | < 1 y | 0.0931 | 0.3 | 0.02 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | > 2 y | 0.095 | 0.3 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifu-gation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample por-tions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 50 | x | Yes | > 2 y | 0.0951 | 0.3 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transi-tions | None | PS-ML | Yes, triphenylphos-phate | Yes-1 | | SB-EUPT | 1 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.0957 | 0.4 | 0.005 | 10 | cold | No | 15 | ACN | Yes, once, citrat buffer, pH 5 | No | Centrifugation, Freezing out, Filtration through syringe filter | | LC-MS/MS QQQ, ESI negativ | | MM-ML | Yes, nicarbazin | No | 104 % (0.2 mg/kg) | SB-EUPT | 3 | Analysis of Phenoxyalkanoic Acids in Milk using QuECgERS method an LC-MS/MS |
| 46 | | No | > 2 y | 0.0961 | 0.4 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extrac-tion solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, 'standard' C18 R.P. chromatog-raphy | None | MM-SL | No | No | | - | | Extraction with ACN+1 % FA. No partitioning step |
| 9 | x | No | None | 0.0967 | 0.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.101 | 0.6 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.088 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 51 | x | Yes | > 2 y | 0.101 | 0.6 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Dispersive-SPE (ODS/ $MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-2 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 3rd-33 | | Yes | > 2 y | 0.100 | 0.6 | 0.02 | 1 | ambient | No | 5 | ACN | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | PS-ML | No | No | 102 % (0.02 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | Yes | > 2 y | 0.103 | 0.7 | 0.01 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 117.1 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | > 2 y | 0.105 | 0.8 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 8 | | Yes | > 2 y | 0.107 | 0.9 | 0.02 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | Yes | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI neg | None | Std. add to extract aliquots | No | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 25‡ | | Yes | > 2 y | 0.108 | 0.9 | 0.02 | 10 | cold | No | 10 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 108.5 % (0.04 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 63 | | Yes | > 2 y | 0.108 | 0.9 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, nicarbazin | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 34 | x | No | > 2 y | 0.109 | 1.0 | | 5 | just thawed | | 10 | EtOAc | Yes, once, HOAc | | Dessication with $Na_2SO_4$, Z-Sep/C18 | | LC-MS/MS QQQ, ES- | LC-MS/MS QQQ, ES- | Std. add to sample portions | | Yes-2 | | SB-EUPT | 1 | |
| 59 | | Yes | > 2 y | 0.112 | 1.1 | 0.01 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation, 1.+150 mg ODS; 2. -15 °C | | | LC-MS/MS QQQ | MM-ML | Yes, 4Cl35DiMeAcedicA | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 30 | | Yes | > 2 y | 0.113 | 1.2 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.113 | 1.2 | 0.02 | 10 | | | 15 | ACN | No | | Freezing out, 2h | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazine | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 66 | x | Yes | > 2 y | 0.189 | 4.6 | 0.02 | 10 | cold | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 47 | x | No | None | 0.390 | 13.8 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 78 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 68 | | No | None | 0.040 | -3.4 | 0.02 | 5 | cold | No | 20 | $H_2O$,ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE (PSA/$MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | - | | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.066 | -3.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 23 | | No | 1 – 2 y | 0.110 | -2.4 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.211 | -1.0 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE (PSA/$MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 36 | x | Yes | 1 – 2 y | 0.216 | -1.0 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE (PSA/$MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | 1 – 2 y | 0.220 | -0.9 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.231 | -0.7 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | No | 1 – 2 y | 0.239 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | 1 – 2 y | 0.244 | -0.6 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE (PSA/$MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.244 | -0.6 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.244 | -0.6 | 0.01 | 10 | ambient | No | 30 | Other, specify under "Solvent details" | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.5 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE (PSA/$MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | None | 0.253 | -0.4 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.257 | -0.4 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 40 | | Yes | 1 – 2 y | 0.265 | -0.3 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 73 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | 1 – 2 y | 0.273 | -0.2 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 22 | | No | 1 – 2 y | 0.274 | -0.1 | 0.1 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 8 | | Yes | 1 – 2 y | 0.277 | -0.1 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, QuEChERS citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |
| 56 | | No | 1 – 2 y | 0.278 | -0.1 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 61 | | Yes | 1 – 2 y | 0.279 | -0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 64 | x | Yes | > 2 y | 0.281 | 0.0 | 0.01 | 2 | cold | No | 1 | $H_2O$,ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylammoniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 57 | | Yes | 1 – 2 y | 0.282 | 0.0 | 0.025 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 90.3 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 34 | x | No | 1 – 2 y | 0.288 | 0.1 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.290 | 0.1 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 58 | x | No | < 1 y | 0.290 | 0.1 | 0.05 | | | | | | | | | | | | | | No | 107 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 3 | | Yes | > 2 y | 0.291 | 0.1 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/$MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 98 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 26 | | Yes | 1 – 2 y | 0.295 | 0.2 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation,only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 66 | x | No | None | 0.295 | 0.2 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 16 | | Yes | < 1 y | 0.296 | 0.2 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 95 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 25 | | Yes | 1 – 2 y‡ | 0.300 | 0.2 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 102.1 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 35 | | Yes | 1 – 2 y | 0.300 | 0.2 | 0.02 | 5 | ambient | No | 10 | $H_2O$,ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltrimethylammonium chloride | No | 104 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 53 | x | No | < 1 y | 0.304 | 0.3 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | 1 – 2 y | 0.309 | 0.4 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-69 | | Yes | 1 – 2 y | 0.310 | 0.4 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, addition of 20 ml of 0.1 % FA solution | No | Freezing out, Centrifuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 112 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |
| 24 | | No | 1 – 2 y | 0.313 | 0.4 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, addition of QuEChERS citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 3rd-71 | | Yes | 1 – 2 y | 0.320 | 0.5 | 0.01 | 2 | ambient | No | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 44 | | Yes | 1 – 2 y | 0.324 | 0.6 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.325 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C12 (Assigned Value = 0.284 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 47 | x | No | None | 0.326 | 0.6 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 74 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 1 | | No | 1 – 2 y | 0.326 | 0.6 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TTP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 7 | x | No | 1 – 2 y | 0.327 | 0.6 | 0.01 | 10 | cold | No | 2 | Other, specify under "Solvent details" | Yes, once | No | Dispersive-SPE, C18/PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 5 | x | Yes | 1 – 2 y | 0.333 | 0.7 | 0.005 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-67 | | No | 1 – 2 y | 0.343 | 0.8 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 93 % (0.5 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 12 | | No | > 2 y | 0.354 | 1.0 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 109.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 49 | x | No | < 1 y | 0.360 | 1.1 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 60 | x | Yes | 1 – 2 y | 0.362 | 1.1 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 48.4 % (48.4 %; CV 6.5 %) | SB-EUPT | 3 | |
| 65 | | Yes | < 1 y | 0.434 | 2.1 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 93 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |
| 2 | x | No | None | 0.543 | 3.7 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 68 | | No | None | 0.046 | -3.3 | 0.02 | 5 | cold | No | 20 | $H_2O$, ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | - | | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.075 | -2.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 23 | | No | 1 – 2 y | 0.117 | -2.3 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 41 | x | No | None | 0.192 | -1.3 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 11 | | Yes | 1 – 2 y | 0.205 | -1.1 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, BAC 12 D6 | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 27 | | No | 1 – 2 y | 0.200 | -1.1 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 7 | x | No | 1 – 2 y | 0.219 | -0.9 | 0.01 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 48 | x | Yes | < 1 y | 0.222 | -0.8 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 31 | x | Yes | 1 – 2 y | 0.223 | -0.8 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.233 | -0.7 | 0.01 | 10 | ambient | No | 30 | Other, not specified | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 36 | x | Yes | 1 – 2 y | 0.240 | -0.6 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | 1 – 2 y | 0.247 | -0.5 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.4 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 61 | | Yes | 1 – 2 y | 0.252 | -0.4 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 22 | | No | 1 – 2 y | 0.262 | -0.3 | 0.1 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 34 | x | No | 1 – 2 y | 0.265 | -0.2 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 8 | | Yes | 1 – 2 y | 0.275 | -0.1 | 0.005 | 5 | just thawed | No | 15 | ACN | Yes, once, QuEChERS citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |
| 66 | x | No | None | 0.277 | 0.0 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 56 | | No | 1 – 2 y | 0.278 | 0.0 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 57 | | Yes | 1 – 2 y | 0.280 | 0.0 | 0.01 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 89.6 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 6 | | Yes | > 2 y | 0.281 | 0.0 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 24 | | No | 1 – 2 y | 0.282 | 0.0 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, addition of QuEChERS citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 40 | | Yes | 1 – 2 y | 0.285 | 0.1 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 77 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 54 | | Yes | 1 – 2 y | 0.286 | 0.1 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 64 | x | Yes | > 2 y | 0.286 | 0.1 | 0.01 | 2 | cold | No | 1 | $H_2O$, ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylammoniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 3 | | Yes | > 2 y | 0.291 | 0.2 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/$MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE (PSA/$MgSO_4$), mechanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 97 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 16 | | Yes | < 1 y | 0.291 | 0.2 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 93 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.292 | 0.2 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | 1 – 2 y | 0.296 | 0.2 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE (PSA/$MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation,only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 47 | x | No | None | 0.297 | 0.3 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 70 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 37 | | No | 1 – 2 y | 0.298 | 0.3 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 25 | | Yes | 1 – 2 y‡ | 0.300 | 0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE (PSA/$MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 100.1 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 44 | | Yes | 1 – 2 y | 0.300 | 0.3 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-71 | | Yes | 1 – 2 y | 0.300 | 0.3 | 0.01 | 2 | ambient | No | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 35 | | Yes | 1 – 2 y | 0.302 | 0.3 | 0.02 | 5 | ambient | No | 10 | $H_2O$, ACN | No | No | Dispersive-SPE (PSA/$MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltrimethylammonium chloride | No | 103 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 5 | x | Yes | 1 – 2 y | 0.308 | 0.4 | 0.005 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE (PSA/$MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.309 | 0.4 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### BAC-C14 (Assigned Value = 0.279 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 12 | | No | > 2 y | 0.315 | 0.5 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 115.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 1 | | No | 1 – 2 y | 0.318 | 0.6 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TTP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.320 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 3rd-67 | | No | 1 – 2 y | 0.323 | 0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | None | MM-ML | Yes, TDCPP | No | 96 % (0.5 mg/kg) | | 4 | QuEChERS – Citrate buffered (EN 15662) |
| 2 | x | No | None | 0.330 | 0.7 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3rd-69 | | Yes | 1 – 2 y | 0.330 | 0.7 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, addition of 20 ml of 0.1 % FA solution | No | Freezing out, Centrifuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 90 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |
| 53 | x | No | < 1 y | 0.335 | 0.8 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | No | < 1 y | 0.340 | 0.9 | 0.05 | | | | | | | | | | | | | | No | 113 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 49 | x | No | < 1 y | 0.350 | 1.0 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 60 | x | Yes | 1 – 2 y | 0.358 | 1.1 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 45.8 % (45.8 %; C.V. = 5.5 %) | SB-EUPT | 3 | |
| 65 | | Yes | < 1 y | 0.878 | 8.6 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 94 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methano
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 66 | x | Yes | > 2 y | 0.090 | -2.0 | 0.02 | 10 | cold | No | 2 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 51 | x | Yes | > 2 y | 0.106 | -1.6 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.116 | -1.4 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 107.5 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 4 | | Yes | < 1 y[#] | 0.118 | -1.4 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 70 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.131 | -1.1 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 22 | | No | > 2 y | 0.137 | -0.9 | 0.02 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 9 | x | No | None | 0.138 | -0.9 | 0.01 | 5 | ambient | No | 45 | MeOH, $H_2O$/HCl | No | No | Freezing out, SPE - column (C18) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, chlormequat chloridD4 | No | | SB-EUPT | 2 | |
| 53 | x | No | < 1 y | 0.144 | -0.8 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.146 | -0.7 | 0.005 | 10 | ambient | No | 5 | MeOH, H2O | | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | DIN EN 15055, 2006-08 |
| 62 | | Yes | > 2 y | 0.150 | -0.7 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 39 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 25 | | Yes | > 2 y[‡] | 0.153 | -0.6 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS | PS-ML | No | No | 107.6 % (0.1 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.158 | -0.5 | 0.02 | 10 | ambient | No | 10 | MeOH : $H_2O$ : HOAc = 75:24:1 | No | No | Centrifugation | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | in house method |
| 58 | x | No | None | 0.160 | -0.4 | 0.01 | 10 | cold | No | 20 | Other, specify under "Solvent details" | No | No | Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | 99 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 3rd-33 | | Yes | 1 – 2 y | 0.160 | -0.4 | 0.02 | 2 | ambient | No | 5 | Other, specify under "Solvent details" | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 77.7 % (0.02 mg/kg) | SB-EUPT | 1 | in house method |
| 38 | x | Yes | > 2 y | 0.164 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 6 | | Yes | > 2 y | 0.168 | -0.2 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | |
| 39 | x | No | < 1 y | 0.170 | -0.2 | 0.02 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | 1 – 2 y[§] | 0.170 | -0.2 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 54 | | No | > 2 y | 0.170 | -0.2 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Clormequat-D4 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; [#] Experience referring to commodity "milk"; [¶] no experience with analytical column; [§] no experience with matrix ofanimal origin; [‡] No experience with milk; [$] Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 55 | x | Yes | > 2 y | 0.170 | -0.2 | 0.02 | 1 | ambient | No | 1 | DCMe, HOAc | Yes, once, add hydroxide ammonium just before clean up | No | Centrifugation, SPE-column (ion exchange), double Centrifugation, Filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, chlormequat D4 | Yes-4 | | SB-EUPT | 2 | in house method |
| 16 | | Yes | < 1 y | 0.172 | -0.2 | 0.01 | 10 | cold | No | 1 | Other, not specified | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | Yes-3 | 95 % (0.143 mg/kg) | SB-EUPT | 3 | Waters Appl. Note |
| 52 | x | No | > 2 y | 0.172 | -0.2 | 0.01 | 20 | ambient | No | 3 | MeOH | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | DIN EN 15055 |
| 8 | | No | > 2 y[§] | 0.175 | -0.1 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 64 | x | Yes | > 2 y | 0.177 | 0.0 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.178 | 0.0 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM, as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 7 | x | No | None | 0.179 | 0.0 | 0.02 | 10 | cold | No | 1 | ACN | No | No | Dispersive-SPE, C18/ PSA | | LC-MS | | Std. add to sample portions | | Yes-2 | | | | |
| 11 | | Yes | > 2 y | 0.179 | 0.0 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 56 | | Yes | > 2 y | 0.179 | 0.0 | 0.004 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, HPLC, Quatrro Premier, ESI+ | None | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 4 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.180 | 0.0 | 0.02 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 35 | | No | None | 0.180 | 0.0 | 0.01 | 5 | ambient | No | 20 | $H_2O$, MeOH | No | No | None | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 44 | | Yes | > 2 y | 0.180 | 0.0 | 0.005 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | §64 LFGB L 00.00-76 |
| 32 | | No | > 2 y | 0.183 | 0.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 59 | | Yes | 1 – 2 y | 0.183 | 0.1 | 0.02 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, Isoproturon-D6 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | > 2 y | 0.186 | 0.2 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.195 | 0.4 | 0.02 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA) ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.197 | 0.4 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; [#] Experience referring to commodity "milk"; [¶] no experience with analytical column; [§] no experience with matrix ofanimal origin; [‡] No experience with milk; [$] Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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 (Assigned Value = 0.179 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 46 | | No | > 2 y | 0.197 | 0.4 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, method QuPPe LC 3 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 1 | | No | > 2 y* | 0.199 | 0.4 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | Yes | > 2 y | 0.199 | 0.4 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 122.1 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 28 | | No | > 2 y | 0.200 | 0.5 | 0.005 | 5 | ambient | | 30 | MeOH | | | Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.205 | 0.6 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 41 | x | No | < 1 y | 0.207 | 0.6 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | Yes | > 2 y | 0.211 | 0.7 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, CCC-D4 | No | | SB-EUPT | 2 | Methanol extraction |
| 47 | x | No | < 1 y | 0.216 | 0.8 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 76 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.216 | 0.8 | 0.02 | 10 | just thawed | No | 20 | Other, Not specified | No | No | Dispersive-SPE, instead of ODS Z-Sep+ was used | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 43 | | No | > 2 y | 0.229 | 1.1 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | > 2 y | 0.240 | 1.4 | 0.01 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 30 | | No | > 2 y | 0.242 | 1.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 63 | | Yes | > 2 y | 0.247 | 1.5 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 27 | | No | > 2 y | 0.279 | 2.2 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | Yes | > 2 y¶ | 0.298 | 2.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 3rd-71 | | Yes | > 2 y | 0.340 | 3.6 | 0.01 | 3 | ambient | Yes, Complete dryness | 10 | Other, specify under "Solvent details" | No | | | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Restek Article |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ¶ no experience with analytical column; § no experience with matrix ofanimal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 32 | | No | > 2 y | 0.097 | -2.6 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 23 | | No | 1 – 2 y | 0.118 | -2.2 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 41 | x | No | None | 0.129 | -2.1 | 0.02 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 34 | x | No | 1 – 2 y | 0.161 | -1.6 | | 10 | just thawed | | 1 | ACN | Yes, once, Citrate salts | | Dispersive-SPE ($PSA/MgSO_4$), Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 54 | | Yes | 1 – 2 y | 0.189 | -1.2 | 0.01 | 10 | ambient | No | 2 | ACN | Yes, once | No | PSA | | | None | PS-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 48 | x | Yes | < 1 y | 0.190 | -1.2 | 0.02 | 5 | cold | No | 10 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), mechanical shaking for 10 min | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | |
| 6 | | Yes | > 2 y | 0.215 | -0.8 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 11 | | Yes | 1 – 2 y | 0.217 | -0.8 | 0.02 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.227 | -0.6 | 0.01 | 10 | ambient | No | 30 | Other, Not specified | No | No | Dessication with $MgSO_4$ and NaCl, Dessication with $MgSO_4$ and NaCl | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | |
| 27 | | No | 1 – 2 y | 0.234 | -0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, TDCP | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | 1 – 2 y | 0.235 | -0.5 | 0.02 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, triphenyl phosphate | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | 1 – 2 y | 0.239 | -0.4 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 64 | x | Yes | > 2 y | 0.239 | -0.4 | 0.01 | 2 | cold | No | 1 | $H_2O$, ACN | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, tetrahexylammoniumbromide | No | | QC | 1 | extraction with $H_2O$-ACN-trifluoraceticacid. Phase separation using sodiumchloride |
| 58 | x | No | < 1 y | 0.240 | -0.4 | 0.05 | | | | | | | | | | | | | | No | 114 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 40 | | Yes | 1 – 2 y | 0.249 | -0.3 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 90 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | 1 – 2 y | 0.250 | -0.3 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 49 | x | No | < 1 y | 0.250 | -0.3 | 0.02 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, Hydro C18 column | LC-MS/MS QQQ | MM-SL | No | No | | - | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 47 | x | No | None | 0.253 | -0.2 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 74 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 8 | | Yes | 1 – 2 y | 0.254 | -0.2 | 0.01 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | No | Yes-2 | | | | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 35 | | Yes | 1 – 2 y | 0.255 | -0.2 | 0.02 | 5 | ambient | No | 10 | $H_2OACN$ | No | No | Dispersive-SPE ($PSA/MgSO_4$), 10 min mechanical shaking | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, Hexadecyltri-methylammonium chloride | No | 103 % (0.1 mg/kg) | QC | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 57 | | Yes | 1 – 2 y | 0.264 | -0.1 | 0.01 | 2 | deep frozen | No | 1 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | No | 91.7 % (0.100 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 61 | | Yes | 1 – 2 y | 0.267 | 0.0 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | MethaNol extraction |
| 3rd-71 | | Yes | 1 – 2 y | 0.270 | 0.0 | 0.01 | 2 | ambient | No | 30 | Other, Not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 37 | | No | 1 – 2 y | 0.272 | 0.1 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, API4000 | None | MM-ML | No | Yes-1 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 2 | x | No | None | 0.276 | 0.1 | 0.02 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 36 | x | Yes | 1 – 2 y | 0.276 | 0.1 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 22 | | No | 1 – 2 y | 0.278 | 0.1 | 0.10 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 7 | x | No | 1 – 2 y | 0.284 | 0.2 | 0.01 | 10 | cold | No | 2 | Other, Not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 5 | x | Yes | 1 – 2 y | 0.285 | 0.3 | 0.01 | 5 | ambient | No | | ACN, $H_2O$ | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ | None | PS-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | 1 – 2 y | 0.289 | 0.3 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifu-gation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 25 | | Yes | 1 – 2 y‡ | 0.289 | 0.3 | 0.02 | 10 | cold | No | 15 | ACN | No | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | PS-ML | No | No | 100.4 % (0.25 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 44 | | Yes | 1 – 2 y | 0.299 | 0.5 | 0.01 | | | | | | | | | | | | | | No | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 13 | | Yes | 1 – 2 y | 0.301 | 0.5 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifu-gation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.301 | 0.5 | 0.01 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 96 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 56 | | No | 1 – 2 y | 0.302 | 0.5 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addi-tion of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.305 | 0.6 | 0.01 | 5 | cold | No | 10 | ACN | Yes, more than twice, citrat buffer, pH 5; PSA/ $MgSO_4$, pH 8; final stabilisation with FA, pH 5 | No | Centrifugation, Dispersive-SPE ($PSA/MgSO_4$), me-chanical shaking | | LC-MS/MS QQQ, ESI positiv | | MM-ML | Yes, Pirimicarb D6, TDCPP | No | 98 % (0.1 mg/kg) | SB-EUPT | 3 | Analysis of Quaternary Ammonium Compounds with modified QuEChERS method; modified: without adding $H_2O$ and shaking |
| 68 | | No | None | 0.306 | 0.6 | 0.02 | 5 | cold | No | 20 | $H_2O$, ACN | Yes, once, after cleanup | No | Freezing out, Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### DDAC-C10 (Assigned Value = 0.268 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 26 | | Yes | 1 – 2 y | 0.310 | 0.6 | 0.01 | 5 | ambient | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, no calculation, only check extraction eff. | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 53 | x | No | < 1 y | 0.310 | 0.6 | 0.02 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 65 | | Yes | < 1 y | 0.313 | 0.7 | 0.02 | 5 | just thawed | No | 2 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, not specified | No | 96 % (0.20 mg/kg) | SB-EUPT | 1 | in house method |
| 1 | | No | 1 – 2 y | 0.320 | 0.8 | 0.02 | 5 | cold | No | 30 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$), Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, TTP | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 24 | | No | 1 – 2 y | 0.321 | 0.8 | 0.02 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 12 | | No | > 2 y | 0.339 | 1.1 | 0.02 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 109.2 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 50 | x | No | > 2 y | 0.343 | 1.1 | 0.02 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, triphenylphosphate | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 60 | x | Yes | 1 – 2 y | 0.349 | 1.2 | 0.02 | 5 | deep frozen | No, None | 10 | ACN | No, None | No, None | Dispersive-SPE ($PSA/MgSO_4$) | | LC-MS/MS QQQ, None | None | MM-SL | No | Yes-1 | 45.6 % (45.6 %; C.V% = 4.5 %) | SB-EUPT | 3 | |
| 3rd-69 | | Yes | 1 – 2 y | 0.400 | 2.0 | 0.01 | 1 | ambient | No | 5 | Salt solution, added acidified $ACN:H_2O$ mix | Yes, once, addition of 20 ml of 0.1 % FA solution | No | Freezing out, Centrifuged for 10 mins at 0 degC | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | 62 % (0.01, 0.1 and 1.0 mg/kg) | SB-Other | 2 | in house method |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 10 | | Yes | > 2 y‡ | <0.01 | -3.8 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 59 | | Yes | 1 – 2 y | 0.012 | -3.7 | 0.01 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, terbuthylazine D5 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | Yes | > 2 y | 0.062 | -2.5 | 0.02 | | | | | | | | | | | | | | No | 41 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 18 | x | Yes | > 2 y | 0.080 | -2.1 | 0.01 | 10 | just thawed | No | 1 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ, Zorbax XDB-C18 | Std. add to extract aliquots | No | Yes-1 | 91.5 % (0.01 mg/kg) | SB-EUPT | 4 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 1 | | No | > 2 y* | 0.094 | -1.8 | 0.01 | 10 | cold | | 30 | ACN | No | No | | | LC-MS/MS QQQ | | MM-ML | Yes, nicarbazim | Yes-5 | | | | QuEChERS for acidic pesticides (EURL-SRM method) |
| 46 | | No | > 2 y | 0.097 | -1.7 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extraction solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, 'standard' C18 R.P. chromatography | None | MM-SL | No | No | | | | Extraction with ACN+1 % FA. No partitioning step |
| 41 | x | No | > 2 y | 0.106 | -1.5 | 0.01 | 10 | ambient | No | 20 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 17 | | No | None | 0.110 | -1.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | Yes-5 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 6 | | Yes | > 2 y | 0.116 | -1.3 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 38 | x | Yes | 1 – 2 y | 0.131 | -0.9 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.132 | -0.9 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |
| 32 | | No | > 2 y | 0.133 | -0.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 52 | x | No | > 2 y | 0.133 | -0.9 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 34 | x | No | > 2 y | 0.137 | -0.8 | | 5 | just thawed | | 10 | EtOAc | Yes, once, HOAc | | Dessication with $Na_2SO_4$, Z-Sep/C18 | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | PS-ML | | No | | SB-EUPT | 1 | |
| 56 | | Yes | > 2 y | 0.137 | -0.8 | 0.00 | 10 | ambient | No | 15 | ACN | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI+ | None | MM-ML | Yes, TPP | No | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 54 | | No | > 2 y | 0.141 | -0.7 | 0.01 | 5 | ambient | No | 20 | EtOAc | Yes, once | No | z-sep+ | | | None | MM-SL | Yes, Pirimicarb-D6 | No | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |
| 27 | | Yes | > 2 y | 0.142 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 31 | x | Yes | > 2 y | 0.150 | -0.5 | 0.01 | 10 | just thawed | | 1 | ACN | | | Dessication with $MgSO_4$, Freezing out | | | | MM-ML | Yes, nicarbazin | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 48 | x | Yes | > 2 y | 0.152 | -0.4 | 0.01 | 5 | cold | No | 10 | ACN | No | No | None | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | | | |
| 8 | | Yes | > 2 y | 0.154 | -0.4 | 0.01 | 5 | just thawed | No | 15 | ACN | Yes, once, citrate-buffer mix | No | Centrifugation, Freezing out | | LC-MS/MS QQQ, ESI pos | None | Std. add to extract aliquots | No | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 11 | | Yes | > 2 y | 0.154 | -0.4 | 0.01 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, nicarbazin | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 64 | x | Yes | > 2 y | 0.154 | -0.4 | 0.01 | 5 | cold | No | 1 | ACN | Yes, once, 1 % HOAc in extraction solvent | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 1 | |
| 23 | | No | 1 – 2 y | 0.157 | -0.3 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 43 | | Yes | > 2 y | 0.158 | -0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | > 2 y | 0.160 | -0.2 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | Yes-5 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.173 | 0.1 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 4 | | Yes | < 1 y# | 0.175 | 0.1 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-Orbitrap | None | MM-ML | Yes, Mecoprop D3 | No | 104 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 22 | | No | > 2 y | 0.175 | 0.1 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, TMA | Yes-3 | | SB-EUPT | 2 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 61 | | Yes | > 2 y | 0.175 | 0.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 47 | x | No | None | 0.184 | 0.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 104 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 3 | | Yes | > 2 y | 0.187 | 0.4 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once, citrat buffer, pH 5 | No | Centrifugation, Freezing out, Filtration through syringe filter | | LC-MS/MS QQQ, ESI negativ | | MM-ML | Yes, nicarbazin | No | 101 % (0.2 mg/kg) | SB-EUPT | 3 | Analysis of Phenoxyalkanoic Acids in Milk using QuECgERS method an LC-MS/MS |
| 5 | x | Yes | < 1 y | 0.187 | 0.4 | 0.01 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 9 | x | No | None | 0.188 | 0.4 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 2 | x | No | > 2 y | 0.189 | 0.5 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | No | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.190 | 0.5 | 0.02 | 10 | cold | No | 15 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 97 % (0.02 mg/kg) | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 62 | | Yes | > 2 y | 0.190 | 0.5 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | 104.4 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | Yes | > 2 y | 0.191 | 0.5 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, nicarbazin | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.194 | 0.6 | 0.01 | 10 | just thawed | No | 15 | ACN | Yes, once, citrate buffer | No | Freezing out, Filtration | | | None | PS-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 25 | | Yes | > 2 y‡ | 0.194 | 0.6 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 107.1 % (0.04 mg/kg) | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 49 | x | No | > 2 y | 0.200 | 0.7 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | None | | LC-MS/MS QQQ, C18 column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 51 | x | Yes | > 2 y | 0.202 | 0.8 | 0.01 | 10 | cold | No | 10 | ACN | No | No | Dispersive-SPE (ODS/ $MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-2 | | SB-EUPT | 2 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 3rd-72 | | No | 1 – 2 y | 0.202 | 0.8 | 0.01 | 10 | cold | No | 1 | ACN | Yes, once | No | Dispersive-SPE | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Fluazifop (Assigned Value = 0.170 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 13 | | Yes | > 2 y | 0.203 | 0.8 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifu-gation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample por-tions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.203 | 0.8 | 0.01 | 25 | ambient | Yes, alkine with NaOH | > 60 | Acetone, CyHn, EtOAc, first hydrolysis | Yes, once | Yes, with tetrabutyl-ammonium-hydroxid/ iosomethane | GPC, Gel-Permeation Chr/phy, acid/base distribution | | | GC-MSD, via second m/z | PS-SL | Yes, d3-Mecoprop, no calculation | No | | SB-EUPT | 1 | alkine hydrolysis extraction GPC, acid/base distribution, methykation,GC-MSD detection |
| 39 | x | No | < 1 y | 0.208 | 0.9 | 0.01 | 10 | | | 15 | ACN | No | | Freezing out, 2h | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazine | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 60 | x | Yes | > 2 y | 0.212 | 1.0 | 0.02 | 10 | deep frozen | No, None | 10 | ACN | No, None | No, None | Freezing out | | LC-MS/MS QQQ, None | None | MM-SL | Yes, nicarbazin | No | 92.5 % (92 %; C.V% 2.8 %) | SB-EUPT | 3 | |
| 3rd-33 | | No | 1 – 2 y | 0.215 | 1.1 | 0.01 | 5 | ambient | No | 5 | QuPPe solvent (MeOH/1 % FA) | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 93 % (0.01 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | > 2 y | 0.217 | 1.1 | 0.01 | 10 | ambient | No | 15 | ACN, $H_2O$ | No | No | Freezing out, Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 30 | | Yes | > 2 y | 0.218 | 1.1 | 0.01 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample por-tions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.225 | 1.3 | 0.01 | 10 | cold | | 1 | ACN | | | Freezing out | | | LC-MS/MS QQQ, 2 transitions | MM-ML | No | No | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 50 | x | Yes | > 2 y | 0.266 | 2.3 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out for 2 hours | | LC-MS/MS QQQ, using 2 transi-tions | None | PS-ML | Yes, triphenylphos-phate | No | | SB-EUPT | 1 | QuEChERS for acidic pesticides (EURL-SRM method) |
| 12 | | Yes | > 2 y | 0.270 | 2.4 | 0.01 | 10 | ambient | No | 30 | ACN | No | No | Centrifugation, Freezing out | | LC-MS/MS QQQ | | PS-ML | No | No | 125.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-71 | | Yes | < 1 y | 0.290 | 2.8 | 0.01 | 3 | ambient | Yes, Complete dryness | 30 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | Yes, nicarbazim | Yes-1 | | SB-EUPT | 2 | Journal of Environmental Science and Health Part B |
| 66 | x | Yes | > 2 y | 0.332 | 3.8 | 0.02 | 10 | cold | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS for acidic pesticides (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Maleic hydrazide (Assigned Value = 0.342 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | No | | FN | -3.4 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | | FN | -3.4 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.192 | -1.8 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 110.5 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 63 | | Yes | 1 – 2 y | 0.229 | -1.3 | 0.05 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 32 | | No | > 2 y | 0.250 | -1.1 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 13 | | Yes | < 1 y | 0.251 | -1.1 | 0.05 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifu-gation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample por-tions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.261 | -0.9 | 0.05 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 53 | x | No | < 1 y | 0.273 | -0.8 | 0.05 | 10 | cold | | 5 | QuPPe solvent | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D2 | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.274 | -0.8 | 0.05 | 10 | ambient | No | 1 | MeOH | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 1 | |
| 38 | x | No | < 1 y | 0.309 | -0.4 | 0.10 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | > 2 y | 0.316 | -0.3 | 0.05 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 111.0 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | No | < 1 y# | 0.327 | -0.2 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 102 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 31 | x | No | < 1 y | 0.337 | -0.1 | 0.10 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 37 | | No | > 2 y | 0.338 | 0.0 | 0.10 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 8 | | No | None | 0.342 | 0.0 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample por-tions | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | No | None | 0.343 | 0.0 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.344 | 0.0 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, maleic hydrazide D2 | Yes-4 | | SB-EUPT | 1 | in house method |
| 47 | x | No | None | 0.346 | 0.0 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample por-tions | Yes, IL-target pesticide | Yes-4 | 102 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 54 | | No | > 2 y | 0.346 | 0.0 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Maleic hydrazide-D2 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Maleic hydrazide (Assigned Value = 0.342 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 46 | | No | None | 0.361 | 0.2 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, QuPPe LC method 2 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | Yes | > 2 y | 0.363 | 0.2 | 0.10 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 5 | x | Yes | < 1 y | 0.369 | 0.3 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 9 | x | No | None | 0.374 | 0.4 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | None | 0.379 | 0.4 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.381 | 0.5 | 0.05 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 21 | | Yes | > 2 y | 0.400 | 0.7 | 0.05 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 2 | x | No | < 1 y | 0.407 | 0.8 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 52 | x | No | None | 0.416 | 0.9 | 0.02 | 10 | ambient | No | 15 | MeOH | Yes, once | No | Liquid-liquid partitioning | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-33 | | Yes | 1 – 2 y | 0.551 | 2.4 | 0.05 | 5 | ambient | No | 5 | QuPPe solvent (MeOH/1 % FA) | Yes, once, Citrate | No | Freezing out | | | LC-MS/MS QQQ | MM-ML | No | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 23 | | No | 1 – 2 y | 0.606 | 3.1 | 0.10 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 22 | | No | None | 0.918 | 6.7 | 0.10 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Note for experience: # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Mepiquat (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 66 | x | Yes | > 2 y | 0.102 | -2.8 | 0.02 | 10 | cold | No | 2 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS-based for milk (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.196 | -1.6 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 106.2 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 51 | x | Yes | > 2 y | 0.219 | -1.4 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 9 | x | No | None | 0.248 | -1.0 | 0.01 | 5 | ambient | No | 45 | MeOH, $H_2O$/HCl | No | No | Freezing out, SPE - column (C18) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | |
| 22 | | No | > 2 y | 0.255 | -0.9 | 0.01 | 10 | ambient | No | 15 | MeOH | Yes, once | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-other substance | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 25 | | Yes | > 2 y‡ | 0.264 | -0.8 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | LC-MS | PS-ML | No | No | 97.0 % (0.1 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 59 | | Yes | 1 – 2 y | 0.273 | -0.7 | 0.02 | 10 | deep frozen | No | 20 | ACN | No | No | Dispersive-SPE (PSA/ $MgSO_4$ + 150 mg ODS), Centrifugation (-15 °C) | | | LC-MS/MS QQQ | MM-ML | Yes, Isoproturon-D6 | Yes-5 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 49 | x | No | 1 – 2 y | 0.280 | -0.6 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.290 | -0.5 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 26 | | Yes | > 2 y | 0.292 | -0.5 | 0.005 | 10 | ambient | No | 5 | MeOH, $H_2O$ | | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | DIN EN 15055, 2006-08 |
| 53 | x | No | < 1 y | 0.294 | -0.5 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D3 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 58 | x | No | None | 0.300 | -0.4 | 0.01 | | | | | | | | | | | | | | No | 65 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 62 | | Yes | > 2 y | 0.300 | -0.4 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 52.5 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-33 | | Yes | 1 – 2 y | 0.300 | -0.4 | 0.02 | 2 | ambient | No | 5 | Other, not specified | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 79.5 % (0.02 mg/kg) | SB-EUPT | 1 | in house method |
| 6 | | Yes | > 2 y | 0.301 | -0.4 | 0.01 | 10 | ambient | No | 1 | MeOH | No | No | Dessication with $Na_2SO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | |
| 37 | | No | > 2 y | 0.301 | -0.4 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Dispersive-SPE (ODS), Filtration, 100 mg ODS/2mL | | LC-MS/MS QQQ, API4000 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | Yes | > 2 y | 0.302 | -0.4 | 0.02 | 10 | ambient | No | 10 | MeOH : $H_2O$ : HOAc = 75:24:1 | No | No | Centrifugation | | LC-MS/MS QQQ | | MM-ML | Yes, Chlormequat C13 | No | | SB-EUPT | 1 | in house method |
| 4 | | Yes | < 1 y# | 0.306 | -0.3 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 108 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; § Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Mepiquat (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 55 | x | Yes | > 2 y | 0.307 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | Yes, chlormequat D4 | Yes-2 | | SB-EUPT | 2 | in house method |
| 38 | x | Yes | > 2 y | 0.311 | -0.3 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.320 | -0.2 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 5 | x | Yes | < 1 y | 0.323 | -0.1 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Mepiquat D3 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 52 | x | No | > 2 y | 0.324 | -0.1 | 0.01 | 20 | ambient | No | 3 | MeOH | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | DIN EN 15055 |
| 54 | | No | > 2 y | 0.326 | -0.1 | 0.02 | 10 | ambient | No | 10 | QuPPe solvent (MeOH/1 % FA) | No | No | Filtration | | | None | PS-ML | Yes, Mepiquat-D3 | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 44 | | Yes | > 2 y | 0.330 | 0.0 | 0.005 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | §64 LFGB L 00.00-76 |
| 63 | | Yes | > 2 y | 0.334 | 0.0 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, IL-target pesticide | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 8 | | No | > 2 y[§] | 0.335 | 0.0 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 1 | | No | > 2 y* | 0.340 | 0.1 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 46 | | No | > 2 y | 0.342 | 0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | Yes, once, extraction solvent contains 1 % HOAc | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, QuPPe LC method 3 | None | PS-SL | Yes, labbeld analogue | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 56 | | Yes | > 2 y | 0.343 | 0.1 | 0.004 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, HPLC, Quatrro Premier, ESI+ | None | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 4 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 16 | | Yes | < 1 y | 0.349 | 0.2 | 0.01 | 10 | cold | No | 1 | Other, not specified | No | No | SPE-column (ion exchange) | | | LC-MS/MS QQQ | PS-ML | No | Yes-3 | 90 % (0.257 mg/kg) | SB-EUPT | 3 | Waters Appl. Note |
| 11 | | Yes | > 2 y | 0.357 | 0.3 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.358 | 0.3 | 0.02 | 10 | just thawed | No | 20 | Other, not specified | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 47 | x | No | < 1 y | 0.359 | 0.3 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 81 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 7 | x | No | None | 0.360 | 0.3 | 0.02 | 10 | cold | No | 1 | ACN | No | No | Dispersive-SPE, C18/PSA | | LC-MS | | Std. add to sample portions | | Yes-2 | | | | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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

### Mepiquat (Assigned Value = 0.333 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3rd-71 | | Yes | > 2 y | 0.360 | 0.3 | 0.01 | 3 | ambient | Yes, Complete dryness | 10 | Other, not specified | No | | | | | LC-MS/MS QQQ | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Restek Article |
| 13 | | Yes | > 2 y | 0.361 | 0.3 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 35 | | No | None | 0.362 | 0.3 | 0.01 | 5 | ambient | No | 20 | $H_2O$, MeOH | No | No | None | | LC-MS/MS QQQ, ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 100 % (0.05 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 24 | | Yes | > 2 y | 0.368 | 0.4 | 0.02 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA) ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | Yes | > 2 y | 0.370 | 0.4 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 118.4 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 31 | x | Yes | > 2 y | 0.375 | 0.5 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, chlormequat | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | > 2 y | 0.377 | 0.5 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 39 | x | No | < 1 y | 0.388 | 0.7 | 0.02 | 10 | | | 10 | QuPPe solvent (MeOH/1 % FA) | No | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | Yes | > 2 y | 0.400 | 0.8 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, CCC-D4 | No | | SB-EUPT | 2 | Methanol extraction |
| 30 | | No | > 2 y | 0.429 | 1.2 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 21 | | Yes | > 2 y | 0.430 | 1.2 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 28 | | No | > 2 y | 0.450 | 1.4 | 0.005 | 5 | ambient | | 30 | MeOH | | | Filtration | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | No | | | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 43 | | No | > 2 y | 0.466 | 1.6 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out | | | None | MM-ML | No | Yes-2 | | SB-EUPT | 1 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | < 1 y | 0.467 | 1.6 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | No | None | 0.472 | 1.7 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 27 | | No | > 2 y | 0.708 | 4.5 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; § no experience with matrix of animal origin; ‡ No experience with milk; $ Experience referring to commodity "Cereal and Feedingstuff"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Chlorate (Assigned Value = 0.185 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 7 | x | No | | FN | -3.6 | 0.1 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | | | | | |
| 62 | | No | | FN | -3.6 | 0.05 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | | | | | QuEChERS – Citrate buffered (EN 15662) |
| 37 | | No | < 1 y | 0.135 | -1.1 | 0.01 | 10 | ambient | No | 20 | MeOH, $H_2O$ | No | No | Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | < 1 y | 0.143 | -0.9 | 0.01 | 10 | ambient | No | 1 | ACN | No | No | Dessication with $MgSO_4$, Centrifugation | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-1 | 0.95 | QC | >5 | |
| 53 | x | No | < 1 y | 0.152 | -0.7 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ion chromatography | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, 18 O 4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.159 | -0.6 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 44 | | Yes | < 1 y | 0.16 | -0.5 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 26 | | Yes | < 1 y | 0.162 | -0.5 | 0.01 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, calculated with delivered ISTD | No | | SB-EUPT | 1 | |
| 47 | x | No | None | 0.172 | -0.3 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-SL | No | No | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 1 | | No | < 1 y* | 0.175 | -0.2 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | Yes | < 1 y# | 0.178 | -0.2 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 103 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.178 | -0.2 | 0.01 | 2 | cold | No | 1 | ACN, $H_2O$ | | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | extraction with ACN/0.1M FA (20/80 v/v) |
| 5 | x | No | < 1 y | 0.180 | -0.1 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, provided by organizer | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.180 | -0.1 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | | |
| 9 | x | No | None | 0.181 | -0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | 1 – 2 y | 0.185 | 0.0 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 50 | x | No | < 1 y | 0.186 | 0.0 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transitions | None | PS-ML | Yes, IL-target pesticide | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 35 | | No | None | 0.187 | 0.0 | 0.02 | 10 | ambient | No | 10 | $H_2O$, QuPPe solvent (MeOH/1 % FA), ACN | No | No | Dispersive-SPE (ODS), 1 min shaking | | LC-MS/MS QQQ, AJS ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 98 % (0.2 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | Yes | < 1 y | 0.195 | 0.2 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Chlorate (Assigned Value = 0.185 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | x | No | None | 0.202 | 0.4 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 48 | x | Yes | None | 0.203 | 0.4 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 30 | | No | None | 0.207 | 0.5 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 31 | x | No | < 1 y | 0.209 | 0.5 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Freezing out | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | < 1 y | 0.210 | 0.5 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 56 | | No | None | 0.235 | 1.1 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI- | None | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 61 | | Yes | 1 – 2 y | 0.236 | 1.1 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, Perchlorate 1804 | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 63 | | Yes | 1 – 2 y | 0.252 | 1.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | < 1 y | 0.276 | 2.0 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | Yes, IL-target pesticide | Yes-1 | 59.6 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Cyromazine (Assigned Value = 0.230 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 28 | | No | > 2 y | 0.097 | -2.3 | 0.01 | 5 | ambient | | 10 | ACN | | | Filtration | | | GC-MS/MS (QQQ) | MM-ML | No | No | | | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 10 | | Yes | > 2 y‡ | 0.109 | -2.1 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 2 | ACN | No | No | Freezing out, Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuEChERS – Citrate buffered (EN 15662) |
| 44 | | Yes | > 2 y | 0.121 | -1.9 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 51 | x | Yes | 1 – 2 y | 0.138 | -1.6 | 0.01 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 18 | x | Yes | > 2 y | 0.145 | -1.5 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Freezing out | | | LC-MS/MS QQQ, Two colums Hypercarb and VDSpher | Std. add to extract aliquots | No | Yes-1 | 101.4 % (0.01 mg/kg) | SB-EUPT | 4 | |
| 6 | | Yes | > 2 y | 0.151 | -1.4 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 3rd-67 | | Yes | > 2 y | 0.163 | -1.2 | 0.01 | 5 | cold | No | 5 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | Yes, PCB 31 | Yes-1 | 36 % (0.1 mg/kg) | SB-EUPT | >5 | QuEChERS – Citrate buffered (EN 15662) |
| 58 | x | No | None | 0.170 | -1.0 | 0.02 | | | | | | | | | | | | | | No | 92 % (0.05 mg/kg) | SB-EUPT | 1 | |
| 8 | | Yes | 1 – 2 y | 0.172 | -1.0 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | LC-MS/MS QQQ, LunaC18 | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 27 | | Yes | > 2 y | 0.179 | -0.9 | 0.01 | 10 | cold | No | 15 | ACN | No | No | Freezing out | | | None | Std. add to sample portions | Yes, TDCP | Yes-2 | | | | QuEChERS – Citrate buffered (EN 15662) |
| 3rd-33 | | Yes | > 2 y | 0.186 | -0.8 | 0.01 | 30 | ambient | No | 5 | EtOAc | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | 99.5 % (0.01 mg/kg) | SB-EUPT | 1 | in house method |
| 62 | | No | > 2 y | 0.190 | -0.7 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | Yes-1 | 60.7 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 41 | x | No | < 1 y | 0.193 | -0.6 | 0.01 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | | LC-MS/MS QQQ | MM-ML | Yes, mepiquat | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 34 | x | No | > 2 y | 0.195 | -0.6 | | 5 | just thawed | | 30 | EtOAc | No | | Filtration + Std. add to sample portions, Filtration, PSA/C18 | | LC-MS/MS QQQ, ES+ | LC-MS/MS QQQ, ES+ | Std. add to sample portions | | Yes-2 | | SB-EUPT | 1 | SweEt type (e.g. T. Pihlström et al. Anal. Bioanal. Chem (200389, 1773-1789) |
| 48 | x | Yes | < 1 y | 0.196 | -0.6 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 63 | | Yes | > 2 y | 0.197 | -0.6 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, trifenylfosfaat | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | < 1 y | 0.214 | -0.3 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 96 % (0.100 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.220 | -0.2 | 0.01 | 10 | cold | No | 1 | ACN | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 55 | x | Yes | > 2 y | 0.221 | -0.1 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, freezingout before Dispersive-SPE (MSPD) + filtration after SPE | | | LC-MS/MS QQQ | PS-ML | No | No | | SB-EUPT | 2 | in house method |
| 4 | | Yes | < 1 y# | 0.224 | -0.1 | 0.01 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 101 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Cyromazine (Assigned Value = 0.230 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 46 | | No | > 2 y | 0.224 | -0.1 | 0.01 | 5 | ambient | No | 30 | 1: ACN + 1 % FA, $H_2O$ | Yes, once, extraction solvent contains 1 % FA | No | None | | LC-MS/MS QQQ, QuPPe LC method 3 | None | MM-SL | No | Yes-3 | | | | Extraction with ACN+1 % FA. No partitioning step |
| 1 | | No | > 2 y* | 0.229 | 0.0 | 0.01 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, IL-target pesticide | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | Yes | > 2 y | 0.248 | 0.3 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 21 | | Yes | > 2 y | 0.250 | 0.4 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 5 | x | Yes | < 1 y | 0.252 | 0.4 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Cyromazine D4 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 50 | x | No | > 2 y | 0.252 | 0.4 | 0.01 | 10 | just thawed | No | 20 | Other, not specified | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 4 transitions | None | PS-ML | No | No | | SB-EUPT | 1 | |
| 64 | x | Yes | > 2 y | 0.253 | 0.4 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 12 | | No | > 2 y | 0.254 | 0.4 | 0.01 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | No | No | 93.5 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 38 | x | No | 1 – 2 y | 0.268 | 0.7 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 66 | x | No | < 1 y | 0.275 | 0.8 | 0.05 | 10 | cold | No | 2 | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuEChERS - Original Version (J. AOAC 86, 2003) |
| 11 | | Yes | 1 – 2 y | 0.278 | 0.8 | 0.01 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.28 | 0.9 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 52 | x | No | > 2 y | 0.285 | 1.0 | 0.01 | 10 | ambient | No | 2 | ACN | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | Yes-1 | | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 32 | | No | > 2 y | 0.287 | 1.0 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 30 | | No | > 2 y | 0.304 | 1.3 | 0.01 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 56 | | No | 1 – 2 y | 0.305 | 1.3 | 0.01 | 10 | ambient | No | 45 | MeOH | Yes, once, addition of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, XEVO, ESI+ | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 24 | | No | < 1 y | 0.307 | 1.3 | 0.01 | 10 | just thawed | No | 15 | QuPPe solvent (MeOH/1 % FA), ACN | Yes, once, by adding acidified MeOH | No | Dispersive-SPE (ODS), Filtration | | | None | Std. add to extract aliquots | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 49 | x | No | 1 – 2 y | 0.320 | 1.6 | 0.01 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | None | | LC-MS/MS QQQ, HILIC column | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 31 | x | No | 1 – 2 y | 0.355 | 2.2 | 0.01 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | | MM-ML | Yes, chlormequat | No | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | 1 – 2 y | 0.361 | 2.3 | 0.01 | 10 | ambient | No | 15 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | | MM-ML | Yes, Carbaryl C13 | Yes-1 | | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"; ‡ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Melamine (Assigned Value = 0.365 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 27 | | No | > 2 y | FN | -3.5 | 1 | 1 | cold | No | 1 | ACN:$H_2O$ = 50:50 | | | SPE, MycoSep (RomerLabs) | | | None | PS-ML | Yes, IL-target pesticide | Yes-3 | | - | 1 | |
| 51 | x | No | None | 0.222 | -1.6 | 0.05 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-69 | | Yes | > 2 y | 0.280 | -0.9 | 0.1 | 10 | cold | No | 30 | ACN, Salt solution, added acidified ACN:$H_2O$ mix | Yes, once, Added 0.5 ml of HCl and 0.5 ml NaOH to adjust pH to 7 | No | Filtration, 0.45um PTFE filter | | | LC-MS/MS QQQ | PS-ML | Yes, 15N3 | Yes-1 | | SB-EUPT | 2 | in house method |
| 3rd-72 | | No | 1 – 2 y | 0.292 | -0.8 | 0.5 | 5 | cold | No | 10 | ACN, other (not specified) | No | No | None | | | LC-MS/MS QQQ | MM-SL | No | No | | SB-EUPT | 1 | |
| 61 | | No | None | 0.305 | -0.7 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 1 | | No | < 1 y | 0.324 | -0.5 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | Yes | > 2 y | 0.340 | -0.3 | 0.05 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 8 | | No | None[‡‡] | 0.347 | -0.2 | 0.01 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA), ACN | No | No | Centrifugation, Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ESI pos | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | | | | |
| 5 | x | No | < 1 y | 0.349 | -0.2 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Melamine tramine 15N3 | Yes-5 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 4 | | No | < 1 y[#] | 0.351 | -0.2 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 92 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 59 | | Yes | > 2 y | 0.360 | -0.1 | 0.1 | 2 | deep frozen | No | 30 | ACN, $H_2O$ | No | Yes, with MSTF at 60 °C | Centrifugation, Filtration | | | GC-MS/MS (QQQ) | MM-SL | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | LNR LABERCA/08MEL-AI.7 |
| 48 | x | Yes | < 1 y | 0.362 | 0.0 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 3rd-33 | | Yes | > 2 y | 0.362 | 0.0 | 0.05 | 1 | ambient | No | 5 | ACN | No | No | None | | | LC-MS/MS QQQ | PS-ML | No | No | 106 % (0.05 mg/kg) | SB-EUPT | 1 | in house method |
| 11 | | No | None | 0.364 | 0.0 | | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | < 1 y | 0.375 | 0.1 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-ML | Yes, IL-target pesticide | Yes-4 | 95 % (0.050 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.375 | 0.1 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 10 | | Yes | > 2 y[‡] | 0.394 | 0.3 | 0.05 | 1 | ambient | No | 5 | ACN | No | No | | | LC-MS/MS QQQ | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | | 4 | |
| 63 | | No | None | 0.394 | 0.3 | 0.05 | 10 | ambient | No | 30 | ACN | | No | Quechers citrate buffer mix, Quechers citrate buffer mix | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | - | 3 | Chlorothalonil: QuEChERS-based mth by EURL-SRM |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: [#] Experience referring to commodity "milk"; [‡‡] Experience only with partcipation in QuPPe Interlaboratory Study [‡] No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Melamine (Assigned Value = 0.365 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 20 | x | Yes | > 2 y | 0.396 | 0.3 | 0.15 | 5 | deep frozen | No | 15 | Diethylamine /$H_2O$/ACN | No | Yes, with BSTFA and 1 % TMCS | Filtration | | GC-MS/MS QQQ | GC-MS/MS (QQQ) | MM-ML | Yes, 2.6 Diamino-4-chloropyrimidine | Yes-1 | | SB-EUPT | 5 | GC-MS Screen for the presence of melamine and related analogs - US FDA |
| 44 | | Yes | > 2 y | 0.396 | 0.3 | 0.25 | | | | | | | | | | | | | | Yes-3 | | SB-EUPT | >5 | in house method |
| 46 | | No | > 2 y | 0.399 | 0.4 | 0.05 | 5 | ambient | No | 5 | ACN | No | No | None | | LC-MS/MS QQQ, HILIC chroma-tograpy | None | PS-ML | Yes, labbeld analogue | Yes-3 | | | | Extraction with ACN |
| 3rd-67 | | Yes | > 2 y | 0.411 | 0.5 | 0.1 | 3 | cold | No | 10 | Other, not specified | No | No | None | | | None | MM-ML | No | No | 107 % (0.5 mg/kg) | | 4 | Technical Specification ISO/TS 15495 IDF/RM230 |
| 21 | | Yes | < 1 y | 0.500 | 1.5 | 0.05 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: ♯ Experience referring to commodity "milk"; ♯♯ Experience only with partcipation in QuPPe Interlaboratory Study ♯ No experience with milk

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Perchlorate (Assigned Value = 0.180 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 32 | | No | > 2 y | 0.050 | -2.9 | 0.01 | | | | | | | | | | | | | | No | | | 1 | |
| 36 | x | No | None | 0.076 | -2.3 | 0.01 | 5 | ambient | No | 10 | MeOH | No | No | None | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 23 | | No | 1 – 2 y | 0.126 | -1.2 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-1 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 6 | | Yes | 1 – 2 y | 0.127 | -1.2 | 0.01 | 10 | ambient | No | 1 | | No | No | Dessication with $Na_2SO_4$ | | LC-MS/MS QQQ | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | | SB-EUPT | 1 | Mini-Luke-Type (Acetone DCM-PE) |
| 7 | x | No | None | 0.132 | -1.1 | 0.02 | 10 | cold | No | 2 | Other, not specified | Yes, once | No | Dispersive-SPE, C18/ PSA | | LC-Q-TOF | | Std. add to sample portions | | Yes-2 | | | | |
| 5 | x | Yes | < 1 y | 0.151 | -0.7 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA), $H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, provided by organizer | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 62 | | Yes | < 1 y | 0.154 | -0.6 | 0.01 | 10 | ambient | No | 2 | ACN, $H_2O$ | No | No | None | | | LC-MS/MS QQQ | PS-ML | Yes, TPP | No | 100 % (0.05 mg/kg) | SB-EUPT | 2 | QuEChERS – Citrate buffered (EN 15662) |
| 53 | x | No | < 1 y | 0.156 | -0.5 | 0.02 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | LC-MS/MS QQQ, ion chromatography | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, 18 O 4 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 37 | | No | < 1 y | 0.158 | -0.5 | 0.01 | 10 | ambient | No | 20 | MeOH, H2O | No | No | Filtration | | LC-MS/MS QQQ, QTRAP5500 | None | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 21 | | Yes | 1 – 2 y | 0.170 | -0.2 | 0.02 | 5 | deep frozen | No | | MeOH | No | No | None | | | | MM-ML | No | No | | | | |
| 12 | | No | < 1 y | 0.171 | -0.2 | 0.02 | 10 | ambient | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Centrifugation | | LC-MS/MS QQQ | | PS-ML | Yes, IL-target pesticide | No | 92.5 % (0.1 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | None | 0.171 | -0.2 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, IL-target pesticide | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.177 | -0.1 | 0.01 | 2 | cold | No | 1 | ACN, $H_2O$ | | No | Oasis-Wax | | | LC-MS/MS QQQ | PS-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | extraction with ACN/0.1M FA (20/80 v/v) |
| 44 | | Yes | < 1 y | 0.180 | 0.0 | 0.01 | | | | | | | | | | | | | | Yes-2 | | SB-EUPT | >5 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | Yes | < 1 y# | 0.181 | 0.0 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 102 % (0.2 mg/kg) | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 9 | x | No | None | 0.184 | 0.1 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-5 | | SB-EUPT | 4 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 26 | | Yes | < 1 y | 0.184 | 0.1 | 0.01 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation | | | LC-MS/MS QQQ | PS-ML | Yes, calculated with delivered ISTD | No | | SB-EUPT | 1 | |
| 35 | | No | None | 0.193 | 0.3 | 0.02 | 10 | ambient | No | 10 | $H_2O$, QuPPe solvent (MeOH/1 % FA), ACN | No | No | Dispersive-SPE (ODS), 1 min shaking | | LC-MS/MS QQQ, AJS ESI pos | None | MM-ML | Yes, IL-target pesticide | No | 104 % (0.2 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 2 | x | No | None | 0.196 | 0.3 | 0.02 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | Yes, Glyphosate C13 | No | | SB-EUPT | 1 | |
| 63 | | Yes | 1 – 2 y | 0.197 | 0.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | Yes, IL-target pesticide | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Perchlorate (Assigned Value = 0.180 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 50 | x | No | < 1 y | 0.198 | 0.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (Z-Sep+) | | LC-MS/MS QQQ, using 2 transi-tions | None | PS-ML | Yes, IL-target pesticide | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 30 | | No | < 1 y | 0.199 | 0.4 | 0.02 | 10 | just thawed | No | 20 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | Std. add to sample por-tions | No | Yes-2 | | | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 11 | | Yes | < 1 y | 0.204 | 0.5 | 0.02 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | Yes | 1 – 2 y | 0.208 | 0.6 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | Yes, Perchlorate 1804 | No | | SB-EUPT | 2 | Methanol extraction |
| 13 | | Yes | 1 – 2 y | 0.210 | 0.7 | 0.01 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample por-tions | Yes, IL-target pesticide | Yes-4 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 31 | x | No | < 1 y | 0.217 | 0.8 | 0.02 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | | | Freezing out | | | | MM-ML | Yes, IL-target pesticide | No | | SB-EUPT | 2 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 56 | | No | None | 0.226 | 1.0 | 0.02 | 10 | ambient | No | 45 | MeOH | Yes, once, addi-tion of FA | No | Freezing out | | LC-MS/MS QQQ, UPLC, Qtrap 5500, ESI- | None | MM-ML | No | Yes-1 | | SB-EUPT | 3 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 10 | | Yes | < 1 y | 0.250 | 1.5 | 0.01 | 10 | cold | Yes, 1.2 ml $H_2O$ | 5 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | >5 | QuPPe-Method for products of plant origin (EURL-SRM method) |
| 1 | | No | 1 – 2 y* | 0.265 | 1.9 | 0.02 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | None | 0.560 | 8.4 | 0.02 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | PS-SL | No | No | | - | | QuPPe-Method for products of animal origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### 4-OH-chlorothalonil (Assigned Value for information only = 0.100 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 61 | | No | None | 0.078 | -0.9 | 0.01 | 10 | deep frozen | No | 1 | MeOH | No | No | Centrifugation, Freezing out, Fitration | | | | PS-ML | No | Yes-1 | | SB-EUPT | 2 | Methanol extraction |
| 13 | | No | None | 0.085 | -0.6 | 0.01 | 10 | cold | No | 15 | ACN | Yes, once | No | Freezing out, Centrifugation | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuEChERS-based for milk (EURL-SRM method) |
| 5 | x | No | < 1 y | 0.092 | -0.3 | 0.01 | 10 | ambient | No | 10 | ACN | No | No | Freezing out | | LC-MS/MS QQQ | None | MM-ML | No | Yes-5 | | SB-EUPT | | QuEChERS – Citrate buffered (EN 15662) |
| 21 | | Yes | > 2 y | 0.103 | 0.1 | 0.01 | 5 | deep frozen | No | | ACN | No | No | Dispersive-SPE ($PSA/MgSO_4$) | | | | MM-ML | Yes, Sulfotep | No | | | | QuEChERS – Citrate buffered (EN 15662) |
| 63 | | No | None | 0.109 | 0.4 | 0.01 | 10 | ambient | No | 30 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | Std. add to sample portions | No | Yes-2 | | - | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | None | 0.111 | 0.4 | 0.01 | 10 | just thawed | No | 15 | ACN | No | No | Freezing out, Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | No | Yes-2 | 86 % (0.100 mg/kg) | SB-EUPT | 1 | QuEChERS-based for milk (EURL-SRM method) |
| 59 | | Yes | 1 – 2 y | 0.119 | 0.8 | 0.01 | 10 | deep frozen | No | 15 | ACN | No | No | Dispersive-SPE (ODS), Centrifugation | | | LC-MS/MS QQQ | MM-ML | Yes, nicarbazin | Yes-5 | | SB-EUPT | 1 | Chlorothalonil: QuEChERS-based mth by EURL-SRM |

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

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
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) | OPTIONAL ANALYTES

### Trimesium (Assigned Value for information only = 0.370 mg/kg)

| Lab-Code SRM9- | NRL | within routine scope | Experience w. analysis of compound | Reported result [mg/kg] | z-Score | RL [mg/kg] | Sample weight [g] | Initial sample temperature | Hydrolysis / Cleavage | Extraction time [min] | Extraction- and/or partitioning solvents | pH-adj. during Extraction / Partitioning | Derivatisation | Cleanup | | Detection | Confirmation | Calibration [1] | ISTD used [2] | Result recovery corrected? [3] | Recovery % (compound. level) | Recovery obtained from [4] | Recovery replicates considered | Method details / Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 26 | | Yes | | FN | -3.5 | 0.05 | 10 | ambient | No | 5 | MeOH | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | PS-SL | No | | | | | |
| 53 | x | No | < 1 y | 0.181 | -0.1 | 0.05 | 10 | cold | | 5 | QuPPe solvent (MeOH/1 % FA) | | | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ, 2 transitions | MM-ML | Yes, D9 | No | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 51 | x | No | None | 0.210 | -1.7 | 0.05 | 10 | cold | No | 10 | MeOH | No | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 4 | | No | < 1 y# | 0.304 | -0.7 | 0.05 | 10 | just thawed | | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | No | No | 106 % (0.2 mg/kg) | SB-EUPT | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 13 | | No | None | 0.320 | -0.5 | 0.05 | 10 | cold | No | 15 | QuPPe solvent (MeOH/1 % FA) | Yes, once | No | Centrifugation, Filtration | | LC-MS/MS QQQ | LC-Q-TOF | Std. add to sample portions | Yes, TPP | Yes-2 | | SB-EUPT | 2 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 38 | x | No | None | 0.350 | -0.2 | 0.02 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Centrifugation, Filtration | | | LC-MS/MS QQQ | MM-ML | No | No | | SB-EUPT | 3 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 64 | x | Yes | < 1 y | 0.354 | -0.2 | 0.01 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | | No | Dispersive-SPE (ODS) | | | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 11 | | Yes | > 2 y | 0.378 | 0.1 | 0.05 | 10 | deep frozen | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration | | LC-MS/MS QQQ | LC-MS/MS QQQ | MM-ML | Yes, IL-target pesticide | Yes-3 | | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 47 | x | No | < 1 y | 0.387 | 0.2 | 0.05 | 10 | just thawed | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS), Filtration (0.45 µm syringe filter) | | | None | Std. add to sample portions | Yes, IL-target pesticide | Yes-4 | 104 % (0.050 mg/kg) | SB-EUPT | 1 | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 48 | x | Yes | < 1 y | 0.387 | 0.2 | 0.05 | 10 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | as proposed by EURL-SRM | | | LC-MS/MS QQQ | Std. add to extract aliquots | Yes, Melamin 15N3 | Yes-4 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 61 | | No | < 1 y | 0.480 | 1.2 | 0.1 | 10 | deep frozen | No | 1 | MeOH | No | No | | | | | PS-ML | No | No | | SB-EUPT | 2 | Methanol extraction |
| 1 | | No | 1 – 2 y* | 0.569 | 2.2 | 0.05 | 5 | cold | No | 1 | QuPPe solvent (MeOH/1 % FA) | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | | MM-ML | No | Yes-5 | | | | QuPPe-Method for products of animal origin (EURL-SRM method) |
| 21 | | Yes | < 1 y | 0.870 | 5.4 | 0.05 | 5 | deep frozen | No | | MeOH, $H_2O$ | No | No | None | | | | MM-ML | No | No | | | | |
| 5 | x | No | < 1 y | 0.914 | 5.9 | 0.05 | 10 | ambient | No | 1 | QuPPe solvent (MeOH/1 % FA)$H_2O$ | No | No | Dispersive-SPE (ODS) | | LC-MS/MS QQQ | None | MM-ML | Yes, Trimethylsulfonium D9 | Yes-5 | | SB-EUPT | | QuPPe-Method for products of plant origin (EURL-SRM method) |

- Abb. of solvent: ACN: acetonitrile; CyHn: cyclohexane; DCM: dichlormethan; FA: formic acid; EtOAc: ethyl acetate; HOAc: acetic acid; MeOH: methanol
- Notes for experience: * very short experience with matrix of animal origin; # Experience referring to commodity "milk"

1) MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition
2) IL : isotopically labelled
3) Yes-1: using recovery figure (as indicated); Yes-2: Yes, aut. via standard add. to sample portions; Yes-3: Yes, aut. via isotope labelled ISTD; Yes 4: Yes, aut. via combination of Yes-2 and Yes-3; Yes-5: Yes, aut. via procedural calibration
4) SB-other: same batch using other matrix ; SB-EUPT: same batch using EUPT-blank matrix; QC: from QC validation data