Ongoing method Performance verification –

Procedural calibration

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O-14 @"Themed day"

FATchers – a novel, simple and fast sample preparation in food of animal origin

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In-house-method „FATchers“

Procedural Standard Calibration

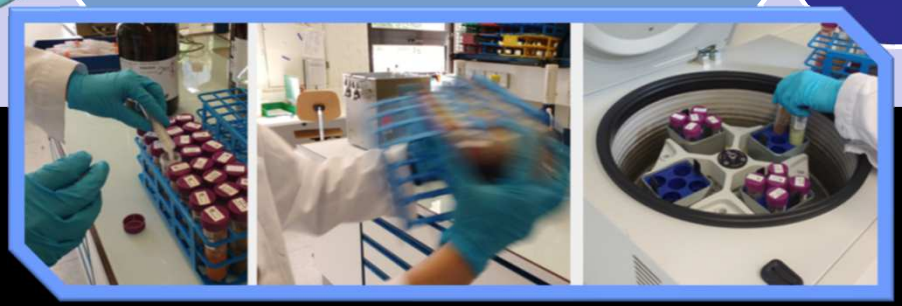
Extraction

- **5g sample** *
- **add 20ml mix ACN:H2O (1:1) + ISTD** – *shake*
- **add 5ml n-Hexan** – shake
- add **Quechers salt 1** #
– shake, centrifuge

 # 450 mg MgSO4, 75 mg CHROMABOND® Diamino

Clean up

- **dispersive SPE - add Quechers salt 2** (600 mg MgSO4, 300 mg CHROMABOND® Diamino, 450 mg CHROMABOND® C18ec)
- – 2 *min shake (Collomix) and centrifuge*



Analysis

- GC-MS/MS (GC-MSD NCI) and/or LC-MS/MS

Optional:

- **Take 2 mL of the eluate** and **evaporate** to dryness
- **Reconstitute in 250µl** ACN (LC) or 250µl Cyclohexane (GC) and inject

* modified method for honey: 10g of sample weight - resolve honey in 10ml water, add 10ml ACN)

FATchers – more than a compromise

Suitable for quite an extended range of **F**at **A**nd **T**ricky matrices

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... plant oils

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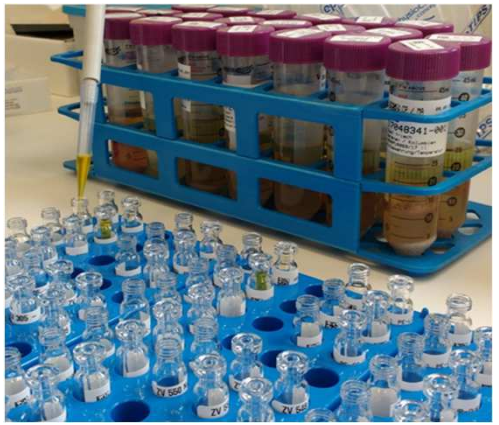
Administration of QC-Samples in AGES Innsbruck

Procedural calibration is an integral part of FATchers method as it **compensates for losses due to liquid/liquid partitioning step**



- Matrix-matched calibration levels prepared by spiking of sample aliquots before sample extraction
- ACN-Extracts are stored in ALS vials over several month
- PC calibration levels are used for each batch of samples, together with at least one freshly prepared QC sample at LOQ-level

Overview – Data set I

GC-MS/MS	plant oils	honey	milk
spike level [mg/kg]	0,02	0,01	0,01
QC sample number(on average)	30	105	45
total number of compounds	370	380	370
compounds within mean apparent recovery 80 - 120 %, RSD ≤20%	99% (79 - 107%)	100% (90 - 111)	99% (77 - 108%)

E4 As a practical approach, residues results do not have to be adjusted for method bias when the mean bias is less than 20% and the default expanded measurement uncertainty of 50% is not exceeded.

Details – Data set I

Percent of QC-samples with apparent recovery **80-120% / < 80% / >120%**

GC-MS/MS	plant oils (30 samples)	honey (105 samples)	milk (45 samples)
Organochlorine			
cis-chlordane	87 / 10 / 3	94 / 1 / 5	92 / 2 / 6
trans-chlordane	94 / 6 / 0	89 / 2 / 9	92 / 3 / 5
endosulfansulfat	88 / 9 / 3	92 / 8 / 0	80 / 6 / 14
gamma-HCH (Lindane)	93 / 7 / 0	82 / 7 / 11	86 / 0 / 14
Hexachlorobenzene (HCB)	100 / 0 / 0	96 / 2 / 2	98 / 2 / 0
Organophosporous compounds			
Chlorpyrifos	94 / 3 / 3	95 / 3 / 2	97 / 0 / 3
Diazinon	91 / 3 / 6	86 / 3 / 11	84 / 3 / 13
Methidathion	87 / 13 / 0	84 / 12 / 4	88 / 6 / 6
Pyrazophos	90 / 10 / 0	81 / 17 / 2	83 / 8 / 9
Pyrethroids			
Bifenthrin	79 / 21 / 0	89 / 9 / 2	85 / 6 / 9
Cypermethrin	79 / 15 / 6	86 / 10 / 4	88 / 3 / 9
Deltamethrin	91 / 9 / 0	80 / 16 / 4	78 / 14 / 8
Permethrin	88 / 8 / 4	80 / 15 / 5	79 / 13 / 8
lambda-Cyhalothrin	84 / 16 / 0	86 / 11 / 3	83 / 10 / 7
Polar (LC-amenable) compounds			
Boscalid	84 / 13 / 3	88 / 10 / 2	86 / 6 / 8
Malathion	88 / 6 / 6	93 / 6 / 1	87 / 6 / 7
Phosmet	80 / 16 / 4	75 / 23 / 2	84 / 14 / 2
Trifloxystrobin	83 / 7 / 10	79 / 14 / 7	92 / 0 / 8

Overview – Data set II

GC-MS/MS	muscle	fish	hens egg
spike level [mg/kg]	0,01	0,01	0,02
QC sample number(on average)	60	45	25
total number of compounds	370	370	370
compounds within mean apparent recovery 80 - 120 % RSD ≤20%	100% (91 - 112)	100% (87 - 115)	99% (73 - 111%)

Details – Data set II

Percent of QC-samples with apparent recovery **80-120% / < 80% / >120%**

GC-MS/MS	muscle (60 samples)	fish (45 samples)	hens egg (25 samples)
Organochlorine			
cis-chlordane	89 / 9 / 2	89 / 2 / 9	93 / 0 / 7
trans-chlordane	89 / 9 / 2	77 / 15 / 8	85 / 0 / 15
endosulfansulfat	65 / 19 / 16	77 / 16 / 7	48 / 38 / 14
gamma-HCH (Lindane)	79 / 12 / 9	83 / 7 / 10	85 / 4 / 11
Hexachlorobenzene (HCB)	98 / 0 / 2	90 / 8 / 2	100 / 0 / 0
Organophosporous compounds			
Chlorpyrifos	94 / 1 / 5	85 / 15 / 0	85 / 0 / 15
Diazinon	86 / 5 / 9	86 / 10 / 4	88 / 0 / 12
Methidathion	79 / 8 / 13	68 / 21 / 11	100 / 0 / 0
Pyrazophos	81 / 12 / 7	76 / 14 / 10	92 / 4 / 4
Pyrethroids			
Bifenthrin	92 / 3 / 5	77 / 6 / 17	96 / 0 / 4
Cypermethrin	82 / 7 / 11	75 / 22/ 3	81 / 8 / 11
Deltamethrin	75 / 16 / 9	71 / 25/ 4	83 / 4 / 13
Permethrin	80 / 13 / 7	70 / 16/ 14	96 / 0 / 4
lambda-Cyhalothrin	67 / 15 / 18	80 / 13 / 7	78 / 9 / 13
Polar (LC-amenable) compounds			
Boscalid	92 / 5 / 3	78 / 4 / 18	92 / 4 / 4
Malathion	80 / 10 / 10	77 / 16 / 7	100 / 0 / 0
Phosmet	69 / 22 / 9	63 / 11 / 26	83 / 13 / 4
Trifloxystrobin	93 / 2 / 5	86 / 0 / 14	88 / 0 / 12

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