



Sveriges lantbruksuniversitet
Swedish University of Agricultural Sciences

Department of Aquatic Sciences
and Assessment



Development of an extraction and clean-up method for suspect and non-target screening of emerging organic pollutants in biota using LC-HRMS

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Aims/Objectives

- ▶ Extraction and clean-up technique for non-target purpose in biota
- ▶ Extraction and clean-up suitable for a broad range of compounds
- ▶ Spiked white-tailed sea eagle muscle tissue for evaluation with 277 compounds
- ▶ Evaluation by matrix effect and recovery

Sampling, extraction, clean-up, fractionation

LC/GC separation and HR-MS analysis

Target analysis

Suspect Screening

Non-target screening

Compound library

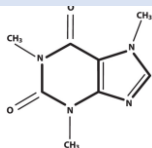
Exact mass filtering

Molecular formula (MS isotope/adducts)

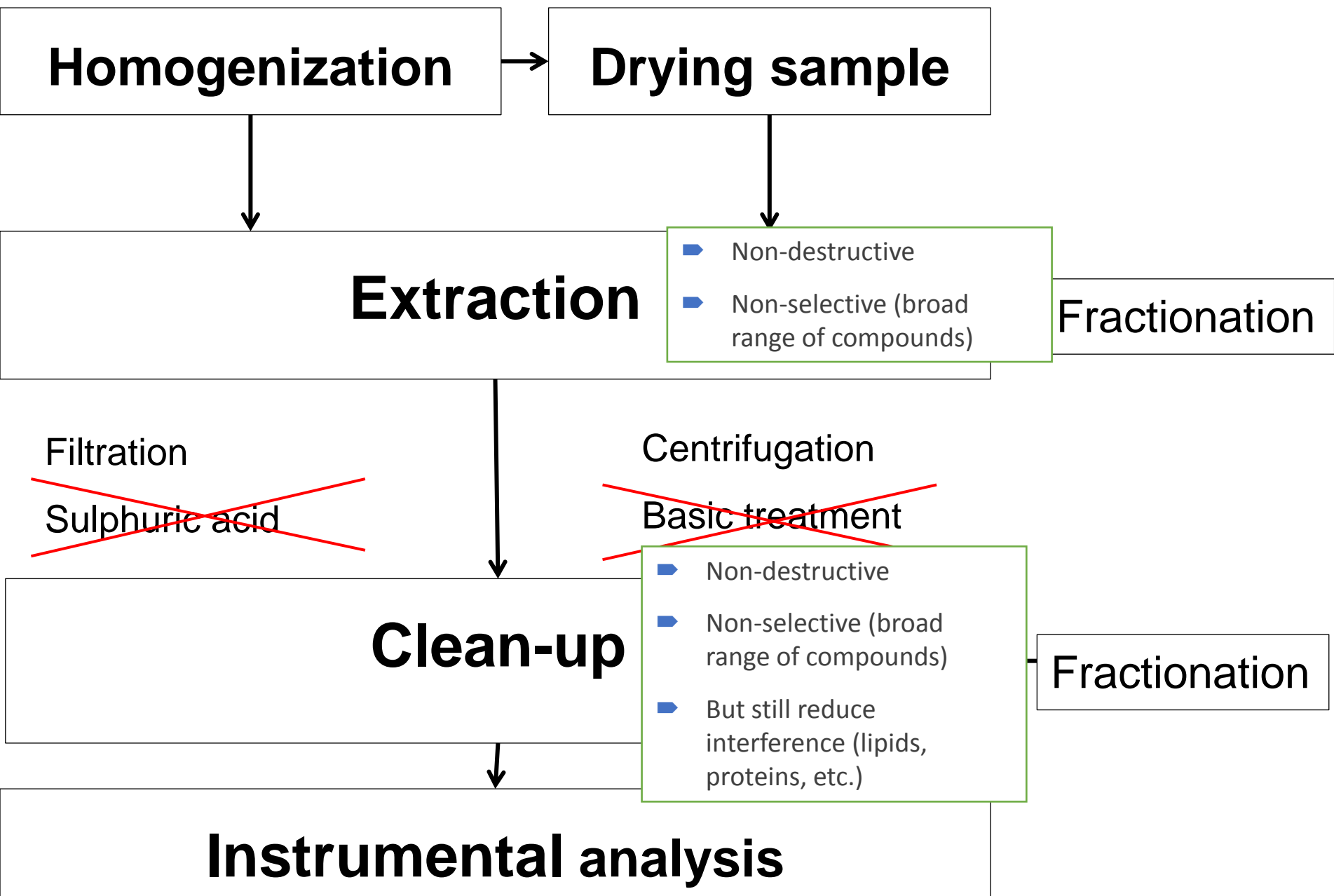
Structure (Database search, MS/MS)

Candidate selection (RT, MS/MS)

Confirmation and quantification (MS/MS, RT, reference standards)



Sample preparation - Biota



Sonication vs QuEChERS



1g White-tailed sea eagle muscle tissue



Homogenization with 5mL ACN

➤ Sonication
(30 minutes)

➤ QuEChERS

➤ 900 mg MgSO₄

➤ 300 mg Z-Sep+

➤ Freezer for >16 hours (denaturalize proteins)

Clean-up SPE (PRiME HLB)

- PRiME HLB (Waters)
 - **P**rocess
 - **R**obustness
 - **I**mprovements in
 - **M**atrix effects
 - **E**ase of use
- Reversed-phase SPE
- 1 mL extract + 4 mL Milli Q
- PRiME HLB
 - Inject extract on cartridges
 - Wash cartridges with 5 mL of 5% MeOH
 - Elute with 5 mL of 100% MeOH



Evaluation of extraction techniques



1g White-tailed sea eagle muscle tissue



Homogenization with 5mL ACN

► **Sonication**
(30 minutes)

► Freezer for >16 hours (denaturalize proteins)

► **SPE**



← spiked 200ng
native + IS

Final extract

► **QuEChERS**

► 900 mg MgSO₄

► 300 mg Z-Sep+

► **SPE**



← spiked 200ng
native + IS

Final extract

► **Matrix effect**



← spiked 200ng
native + IS

Solvent blank

Matrix Effect > 1 -> Matrix enhancement

Matrix Effect < 1 -> Matrix suppression

Evaluation of extraction techniques



spiked 200ng
native + IS

➤ Recovery



spiked 200ng
native + IS

Solvent blank = 100 %

1g White-tailed sea eagle muscle tissue



Homogenization with 5mL ACN

➤ Sonication

(30 minutes)

➤ Freezer for >16 hours (denaturalize proteins)

➤ SPE



Final extract

➤ QuEChERS

➤ 900 mg MgSO₄

➤ 300 mg Z-Sep+

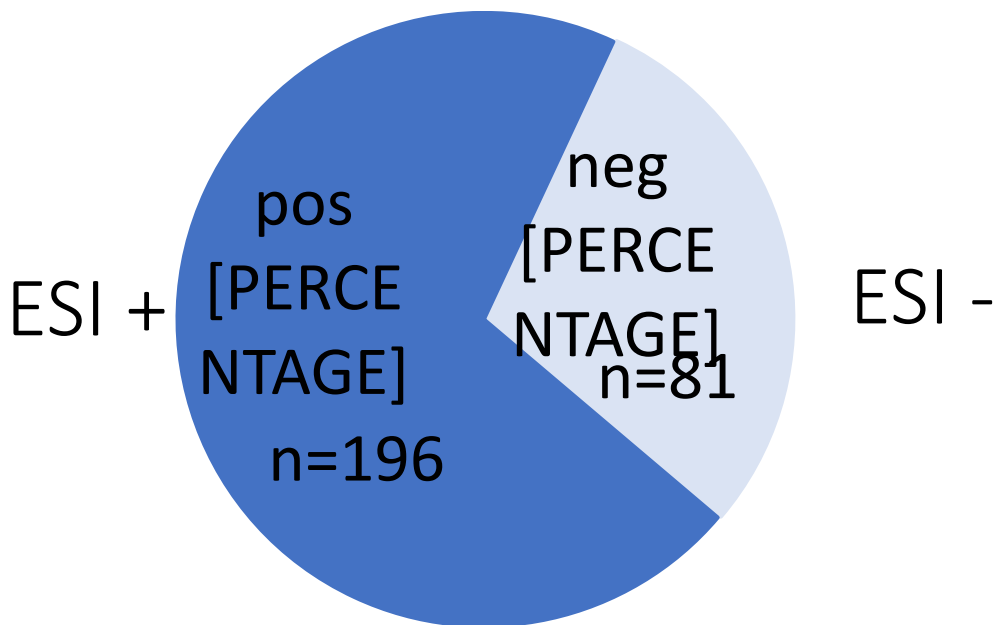
➤ SPE



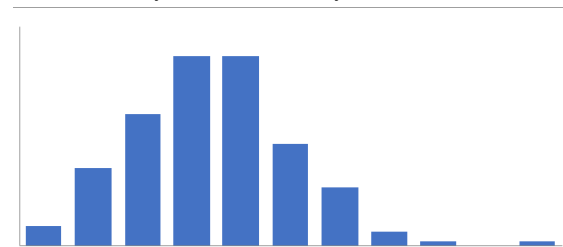
Final extract

Results

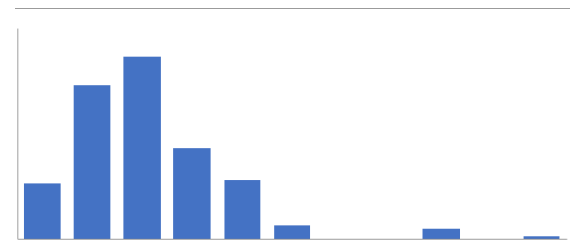
Compounds (n= 277)



- Log K_{ow} :
 - -2,00 till 10,43



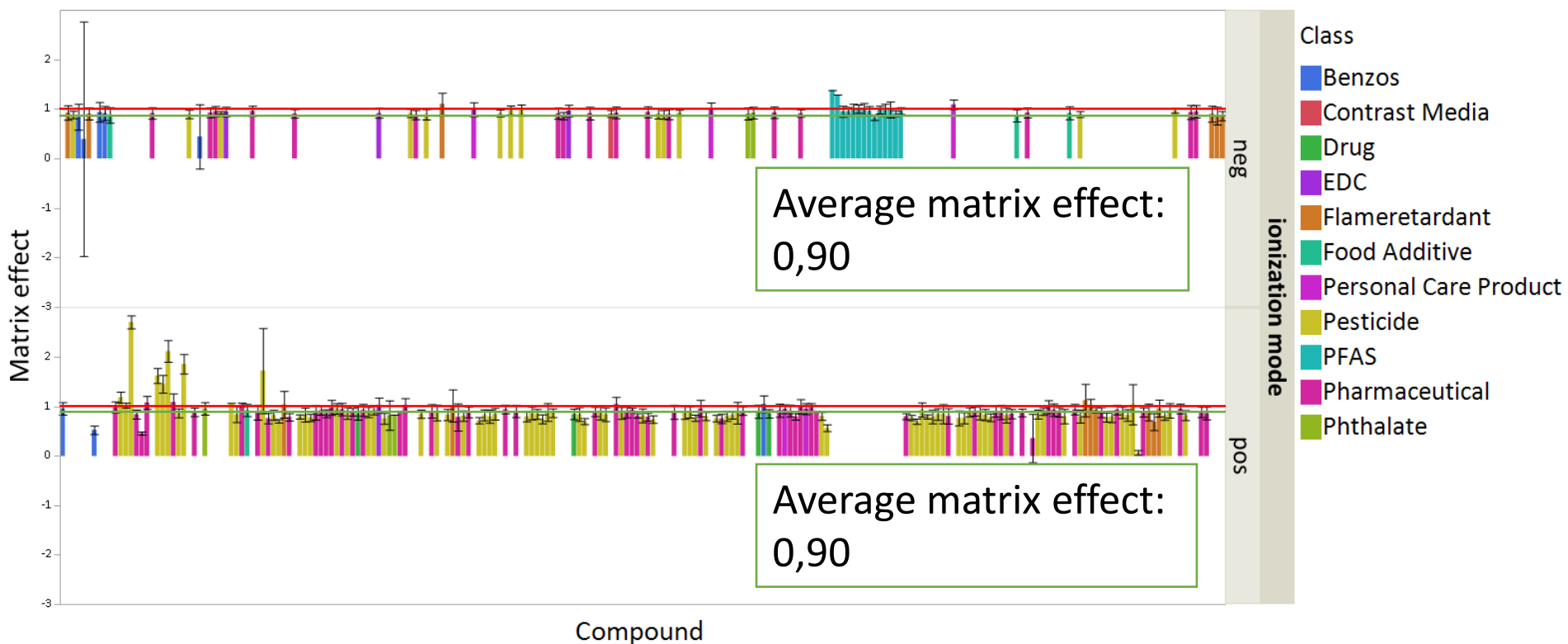
- Molecular weight:
 - 119,127 till 837,047



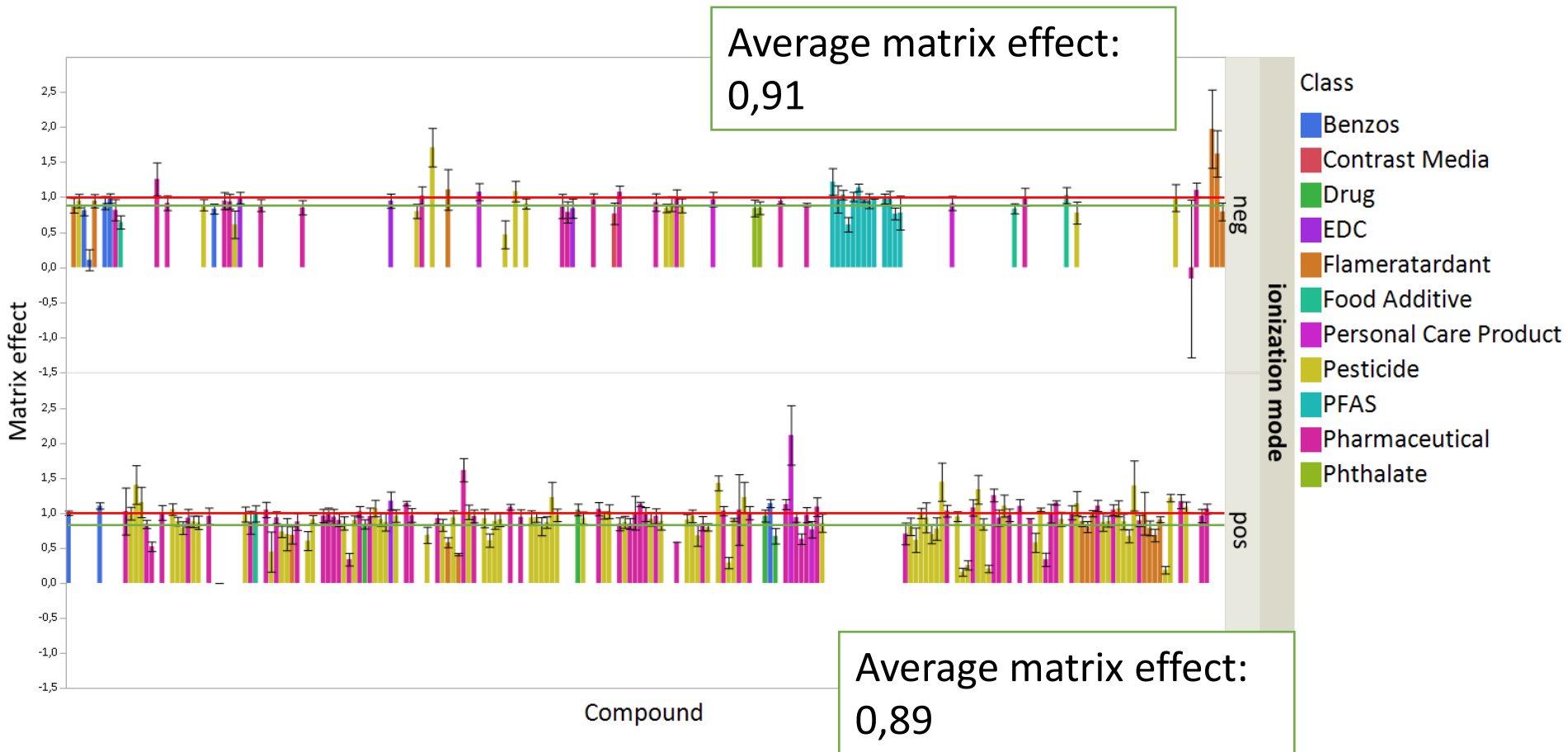
➡ Detected compounds

- Benzotriazoles/ Benzothiazoles (n= 7)
- Contrast Media (n=1)
- Drugs (n=4)
- Endocrine Disrupting Compounds (n=4)
- Food Additives (n=4)
- Flame retardants (n=17)
- Personal Care Products (n=5)
- Pesticides (n=90)
- PFAS (n=14)
- Pharmaceuticals (n=77)
- Phthalates (n=3)

QuEChERS + SPE: Matrix effect for all compounds

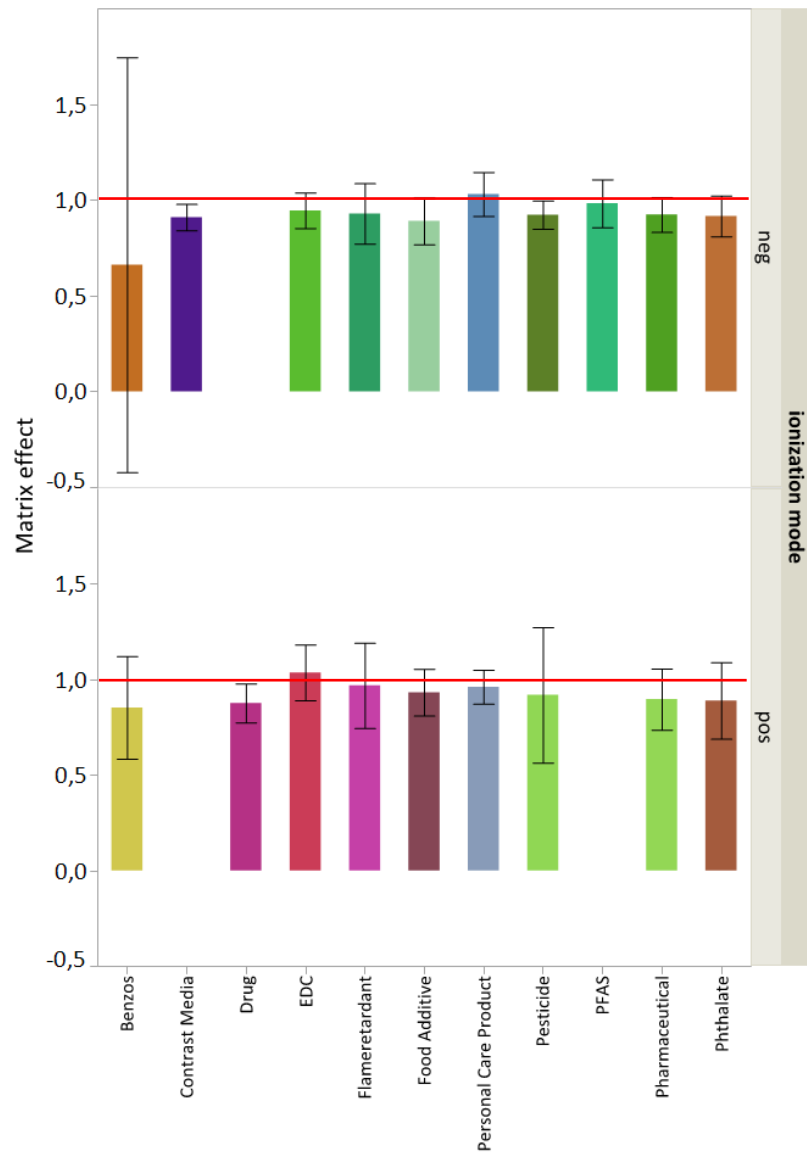


Sonication + SPE: Matrix effect for all compounds

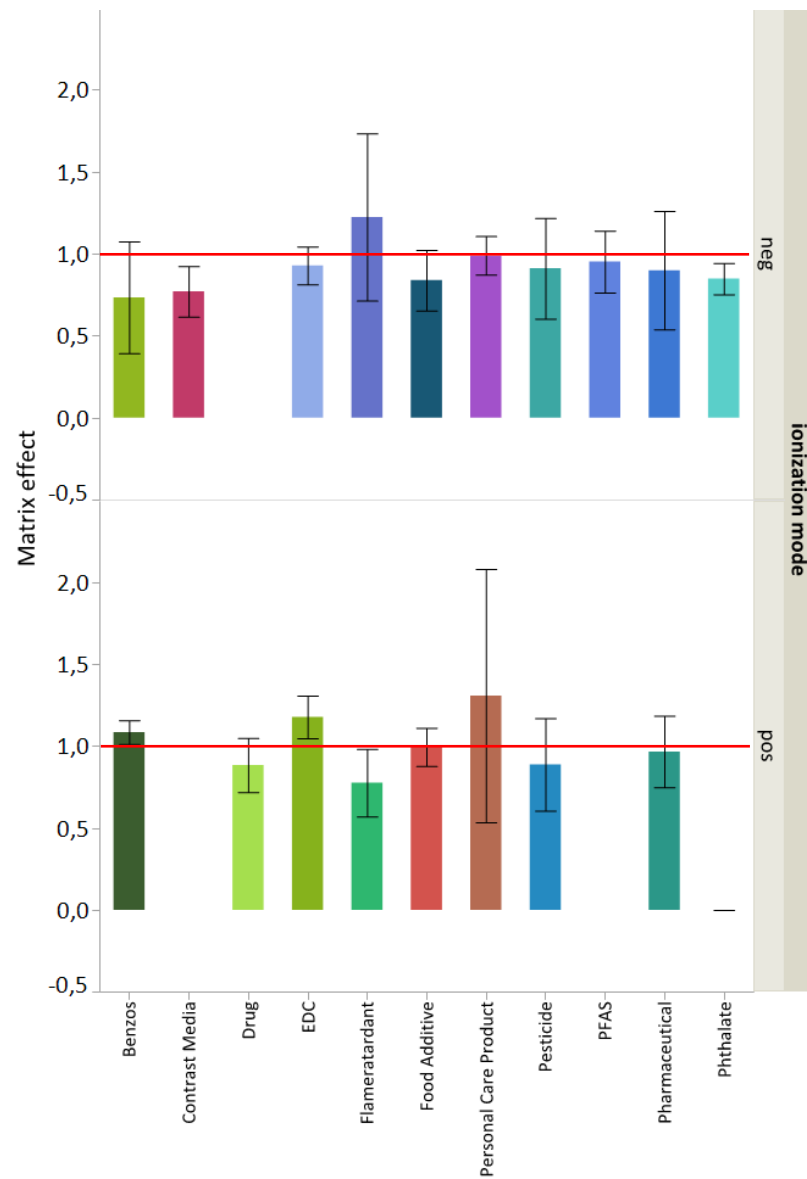


Matrix effect for each compound class

QuECHERS



Sonication

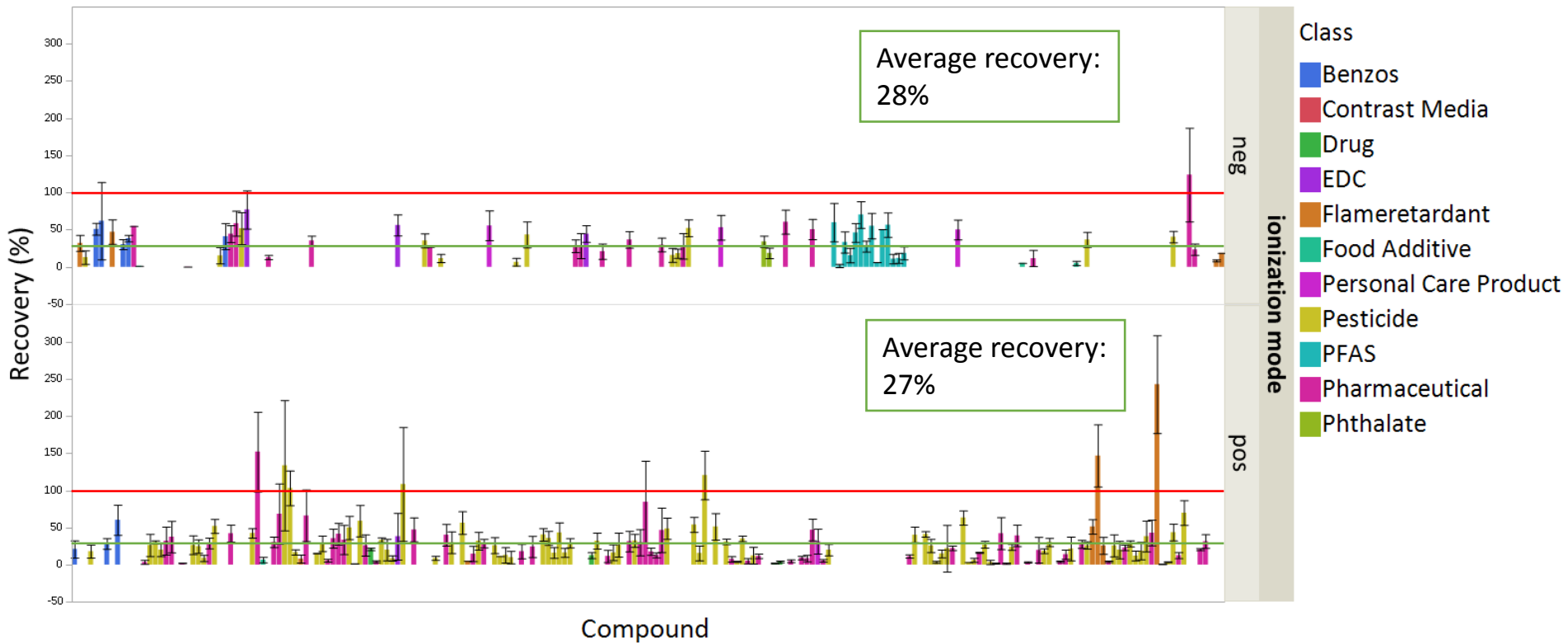


QuEChERS + SPE: % Recovery for all compounds



Sonication + SPE:

% Recovery for all compounds



Conclusion

- ▶ Matrix effect: low for both extraction methods
- ▶ Recovery: poor, but better for sonication extraction than for QuEChERS
- ▶ Future:
 - ▶ of recovery by another clean-up technique, like GPC
 - ▶ Investigate losses

Acknowledgement

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-
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Naturhistoriska
riksmuseet