



# recovery of humic substances from ion exchange brine

Elisabeth Vaudevire

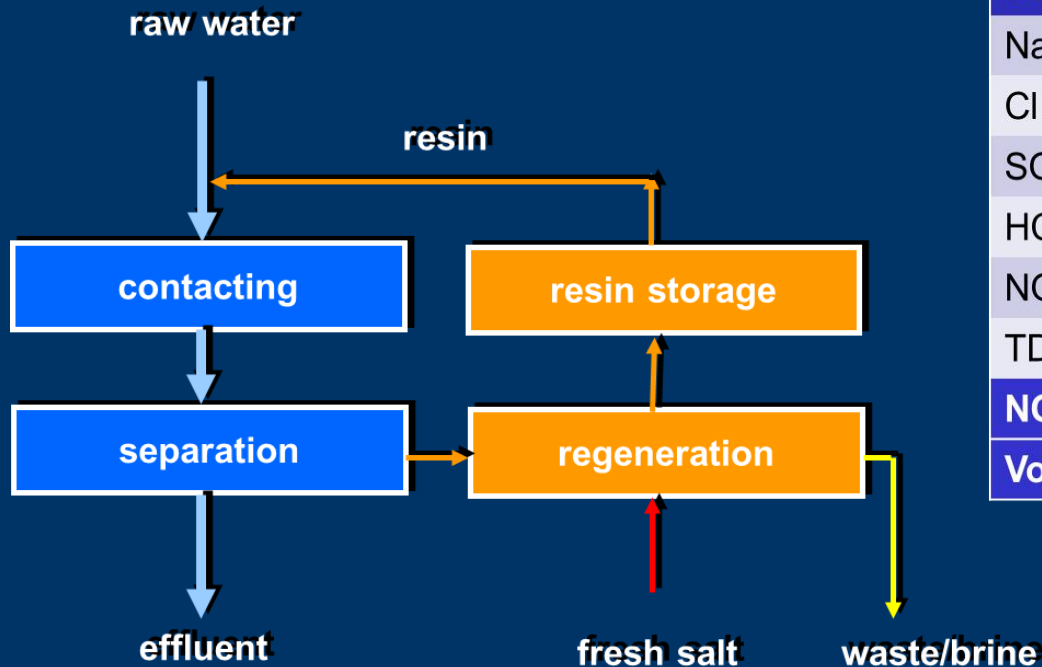
# recovery of humic substances from ion exchange brine

## overview

- origin of the brine – specific adsorption of humic substances by IEX
- characterization of the humic substances
- technological aspects for compounds separation and recovery
- contamination with micropollutants
- applications opportunity for humic substances, business case and legal framework

# origin and quality figures

## IEX process

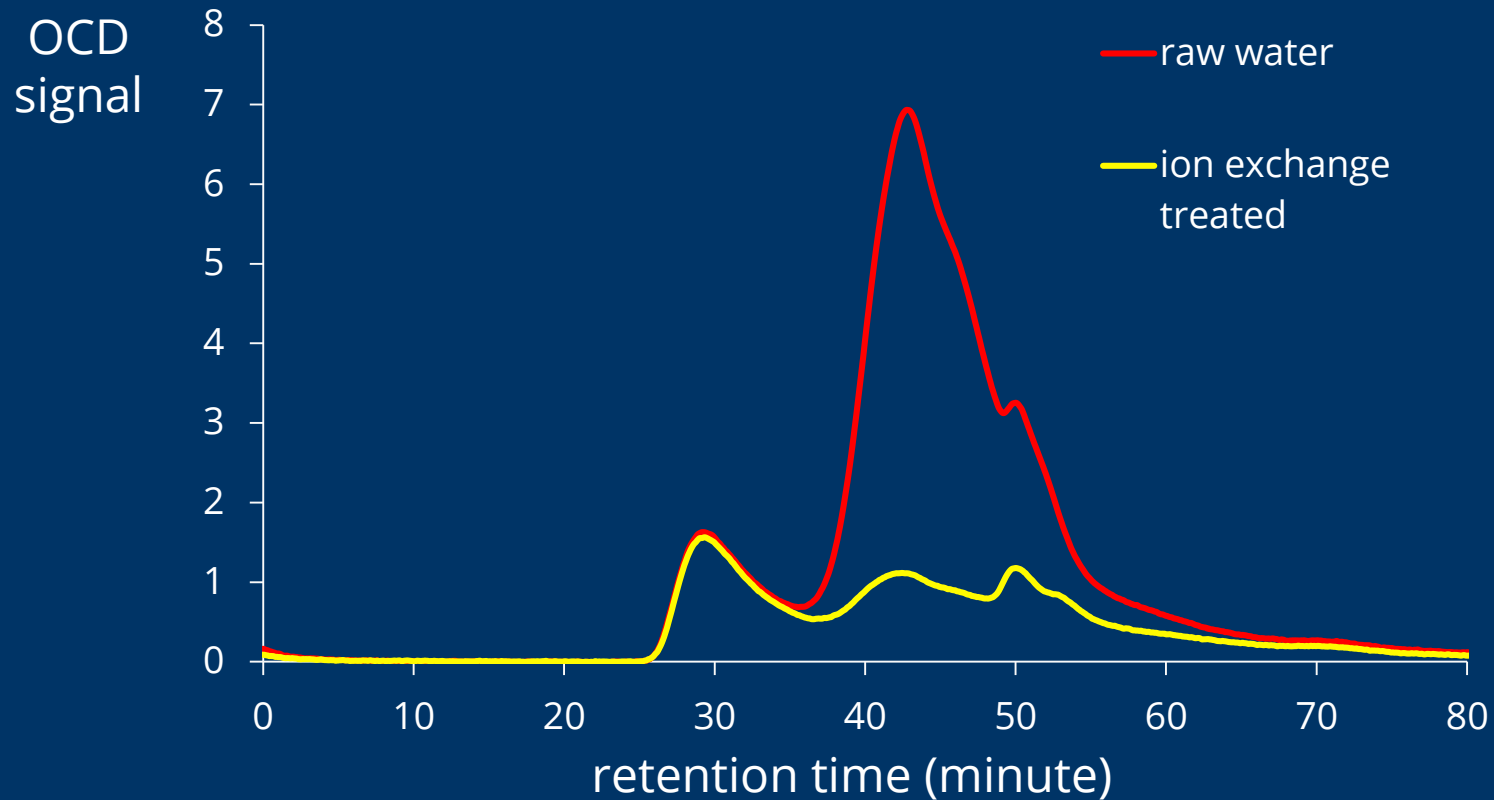


Water soluble ions	SIX® regenerant solution
Na	15 g/L
Cl	10 g/L
SO <sub>4</sub>	8 g/L
HCO <sub>3</sub>	4 g/L
NO <sub>3</sub>	0,2 g/L
TDS	
NOM	0,5 g/L
Volume	10-25 m <sup>3</sup> /h



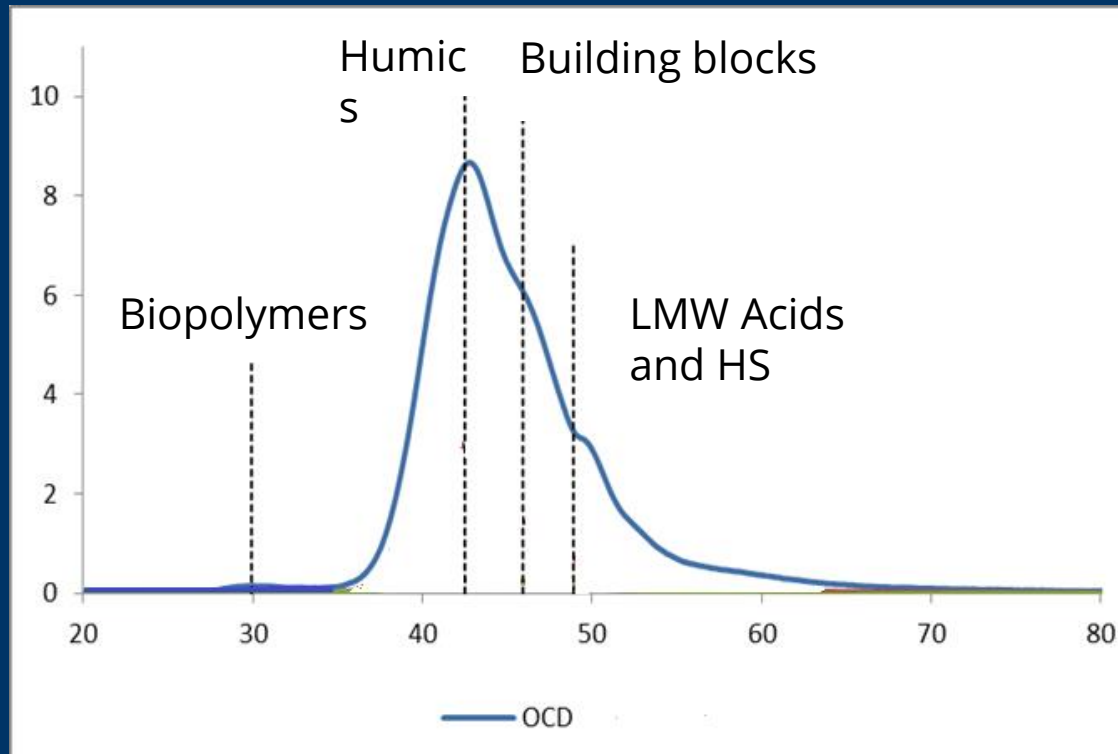
# NOM adsorption to IEX resin

## LC OCD profile



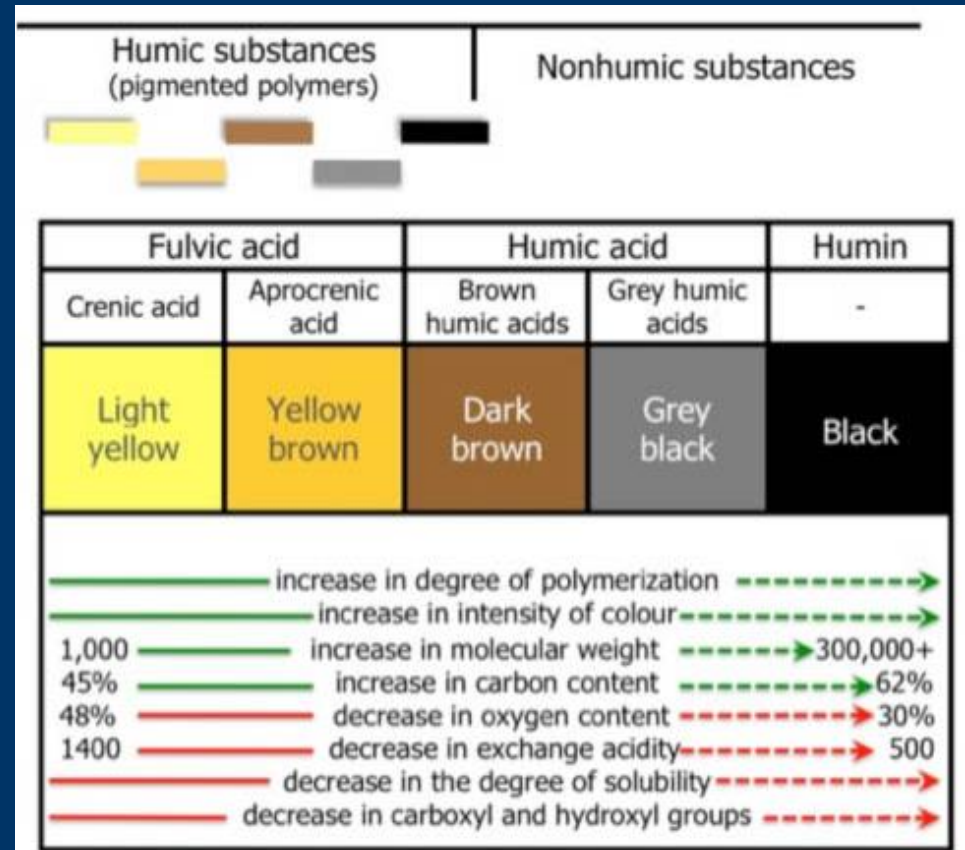
# NOM desorbed in the brine

## LC-OCD profile



# characterization of the humic substances

- humic fraction as part of NOM
- Why characterization
- Prior step of purification
- Results from Fourier transform infra red characterization



# characterization of the humic substances

isolation protocol recommended by IHSS



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DI UDINE

- acidification to pH 1 to 2
- precipitate neutralized, centrifuged and reacidified until inorganic ashes <1% = humic fraction
- supernatant passed through XAD-8 column = fluvic fraction





# characterization of the humic substances

isolation protocol recommended by IHSS

SAMPLE	Corg mg/L	Humic C	fractions
IEX brine PWN	total NOM	2112	
	non humic C	1070	52% humic acid
	humic C	1093	49% fluvic acid
IEX brine DEWG	total NOM	10409	
	non humic C	4707	48% humic acid
	humic C	4978	63% fluvic acid
Waste Water treated	total NOM	1328	
	non humic C	1079	24% humic acid
	humic C	323	43% fluvic acid



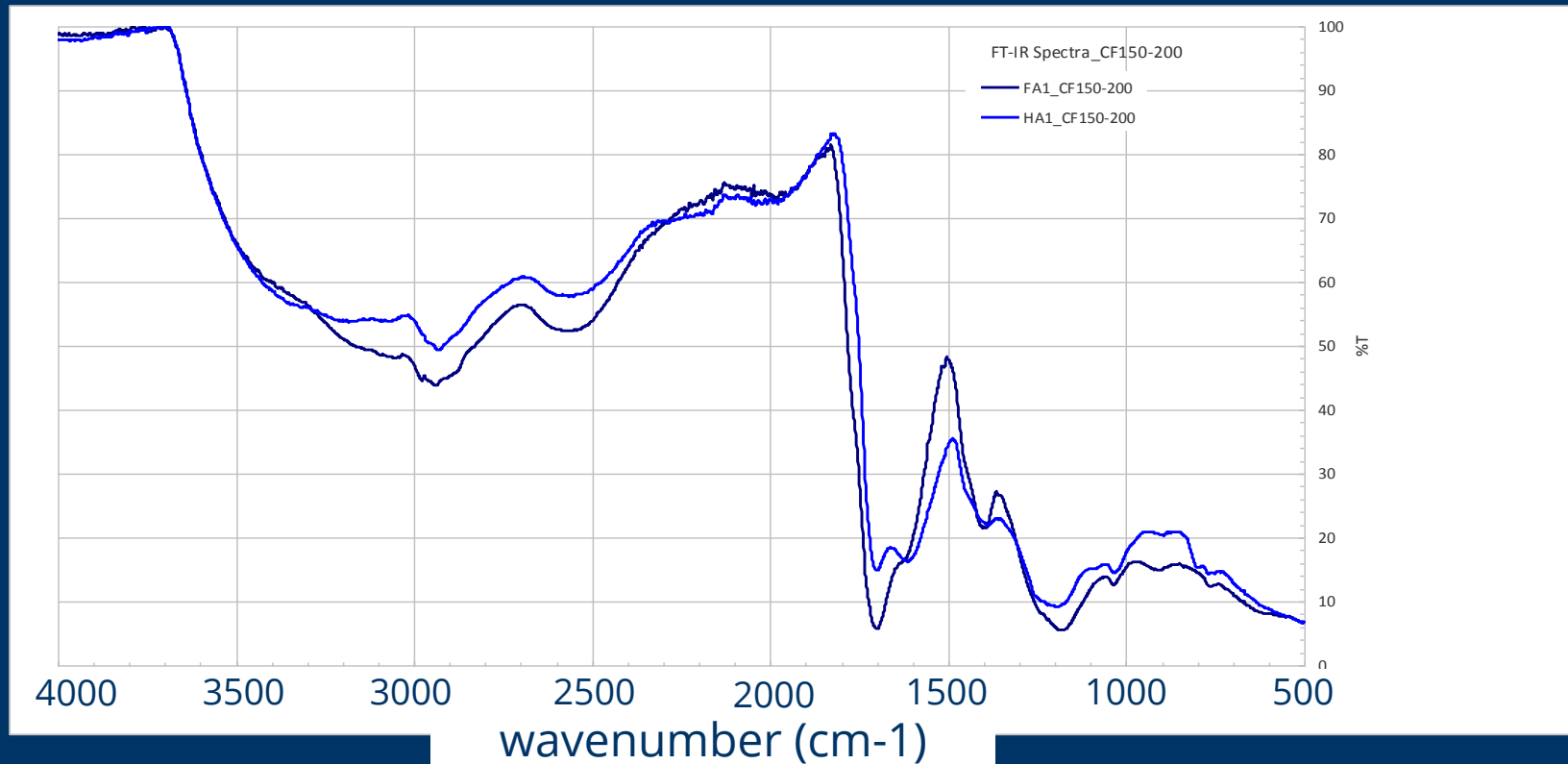
# characterization of the humic substances

## FTIR spectra

- Fourier transform infra red spectrometry:
  - qualitative molecular analysis method
  - use infra red source as baseline of energy
  - when a sample is expose to IR, absorbance of that sample is used to measure the bonds of the carbon atoms

# characterization of the humic substances

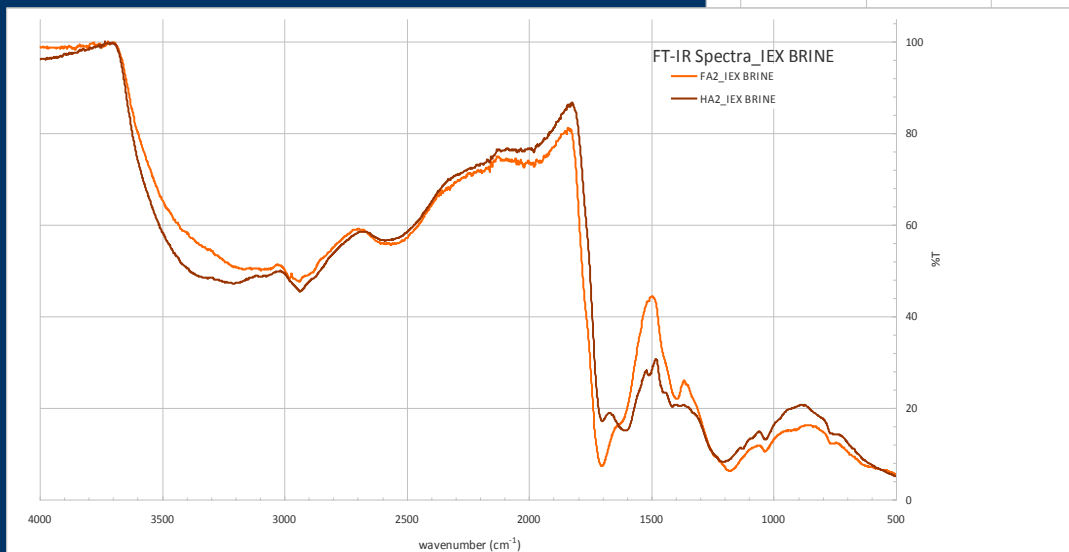
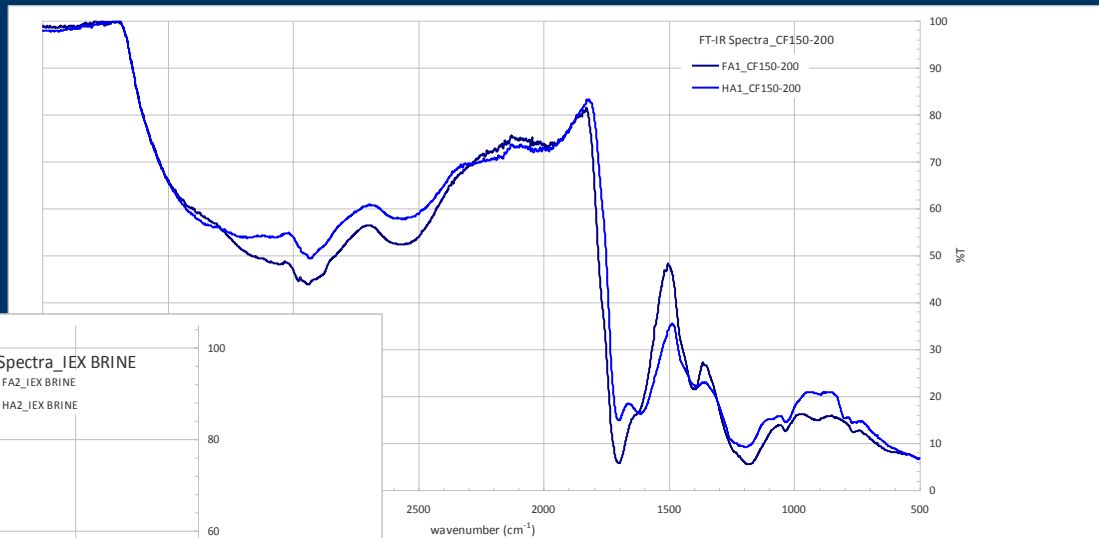
## FTIR spectra



# characterization of the humic substances

FTIR spectra

PWN



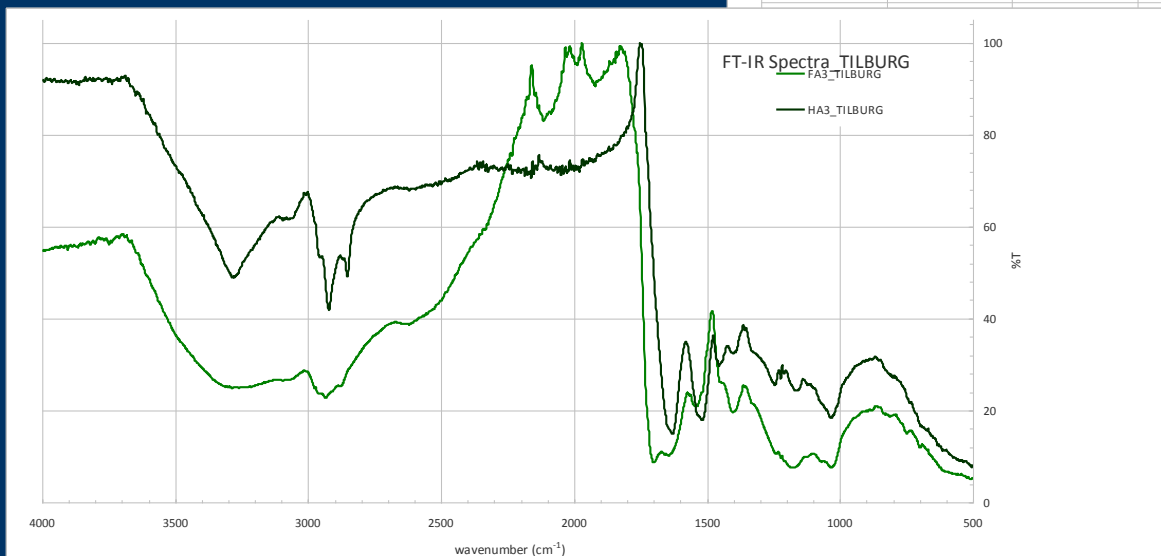
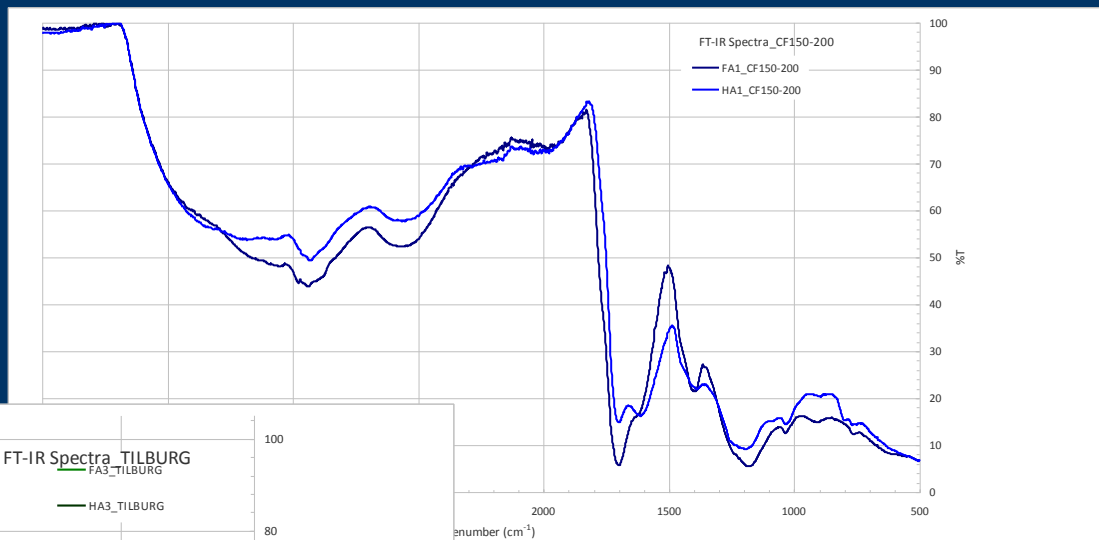
de Watergroep

**SIMILAR CARBON BONDS**

# characterization of the humic substances

FTIR spectra

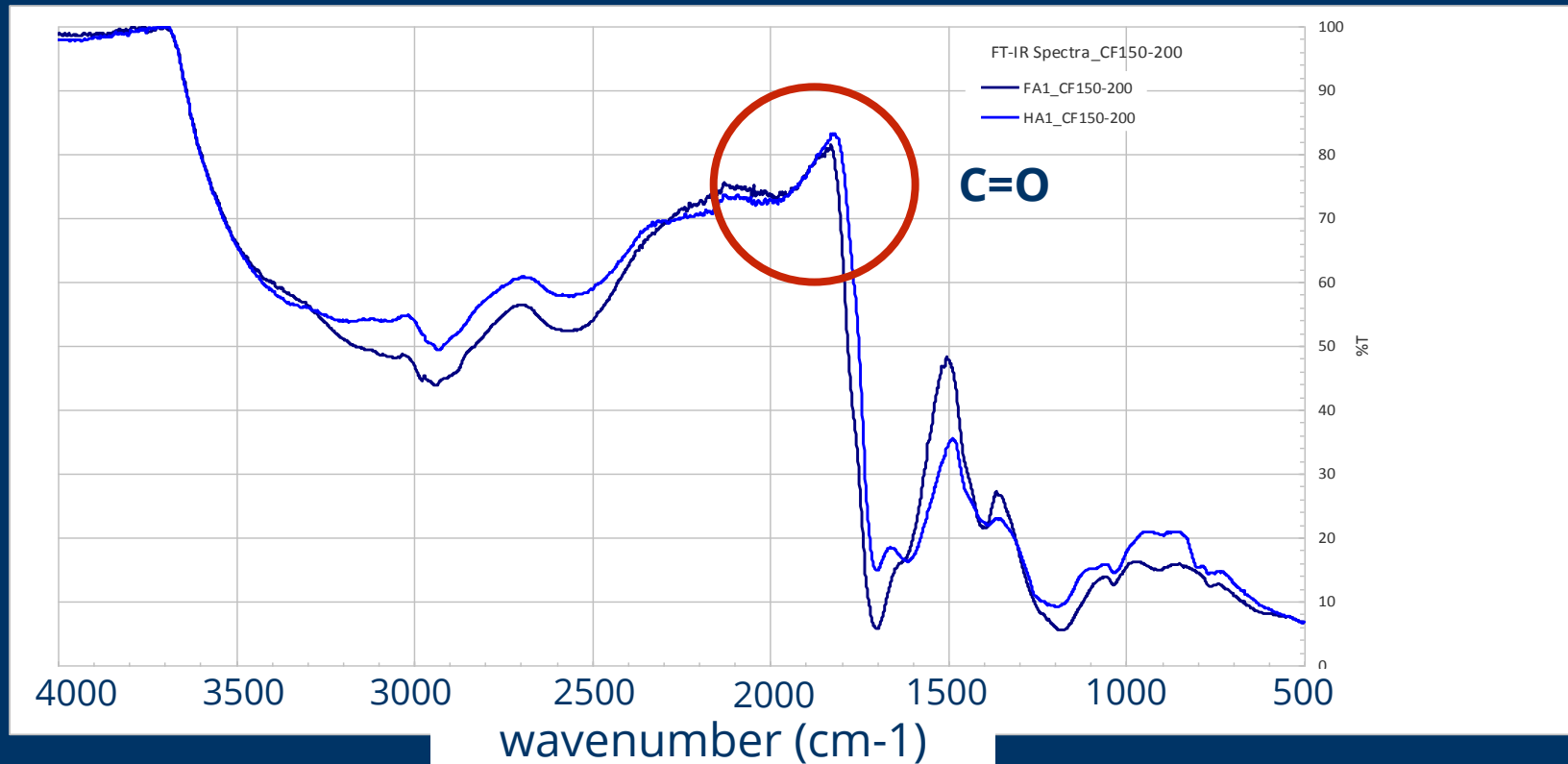
PWN



waste water  
evides

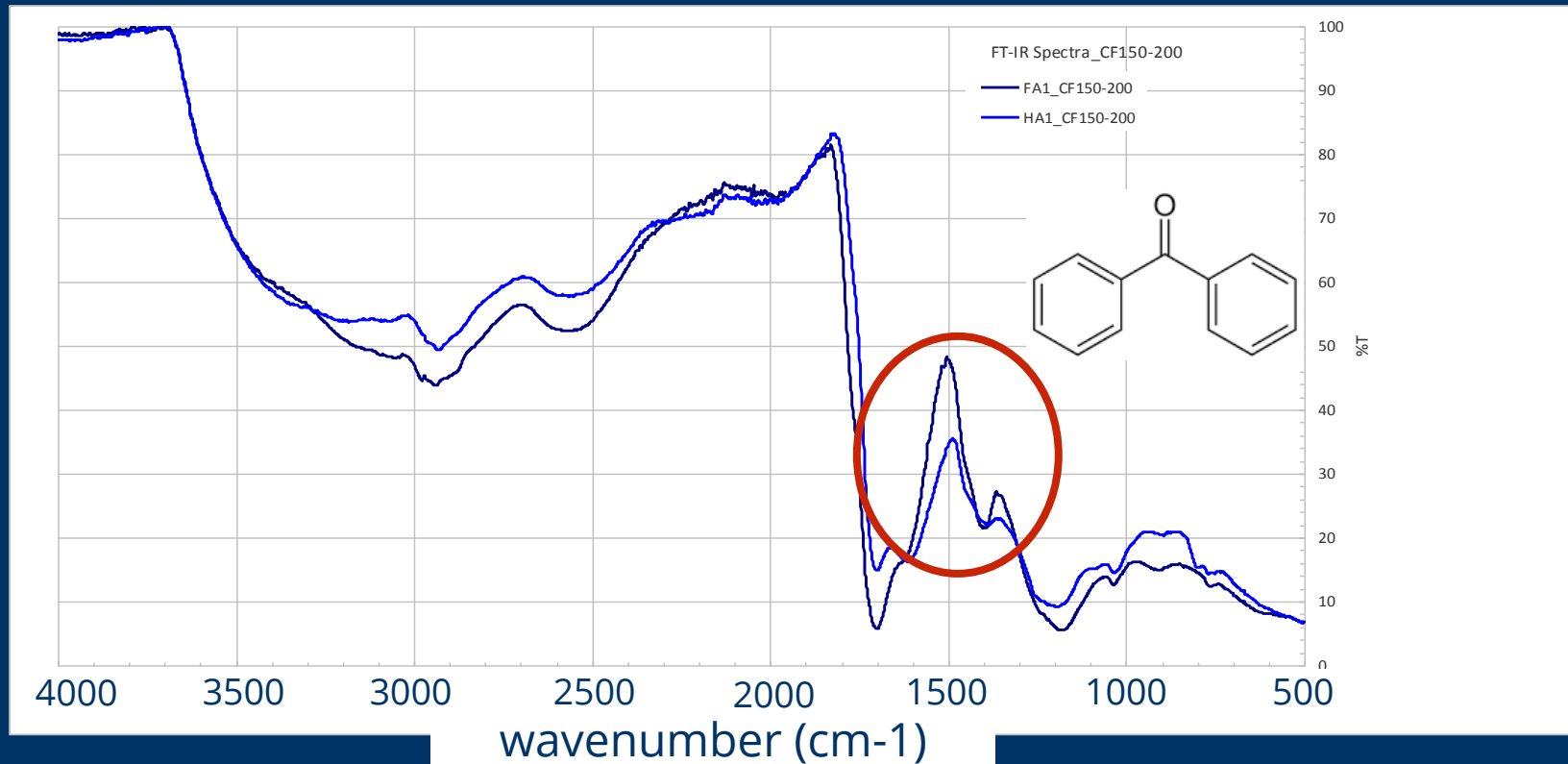
# characterization of the humic substances

## FTIR spectra



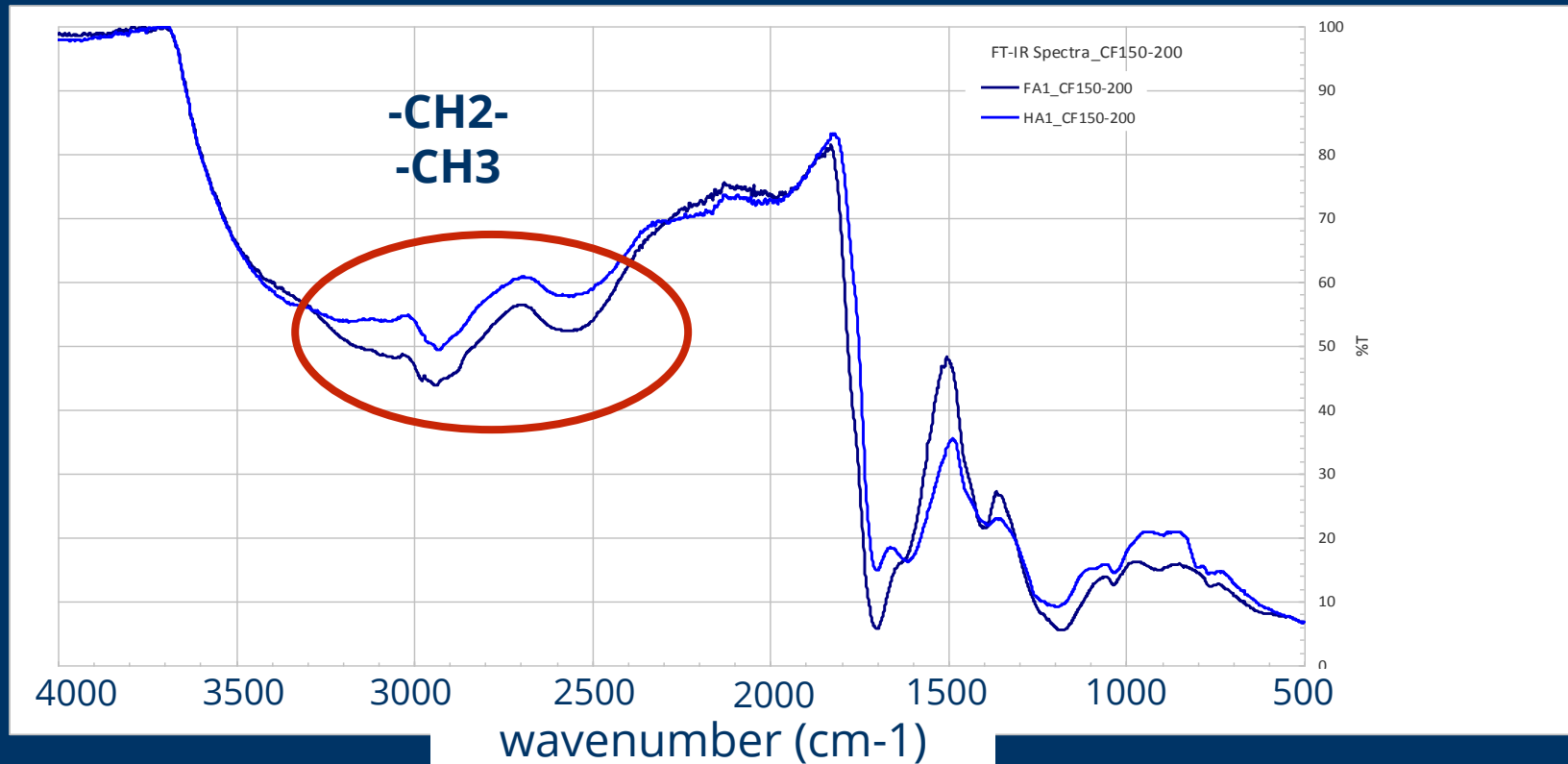
# characterization of the humic substances

## FTIR spectra



# characterization of the humic substances

## FTIR spectra





# characterization of the humic substances

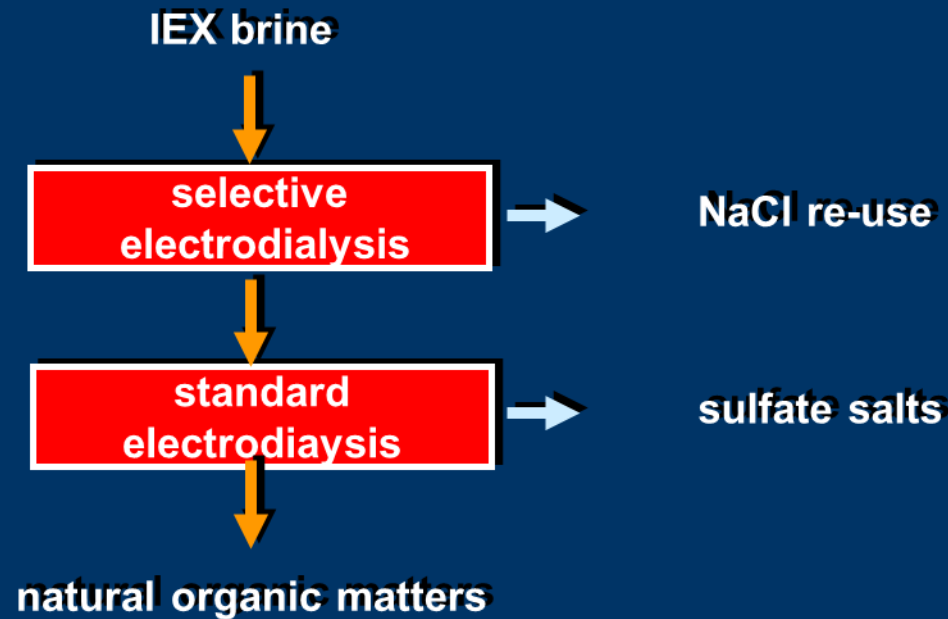
## FTIR – conclusions



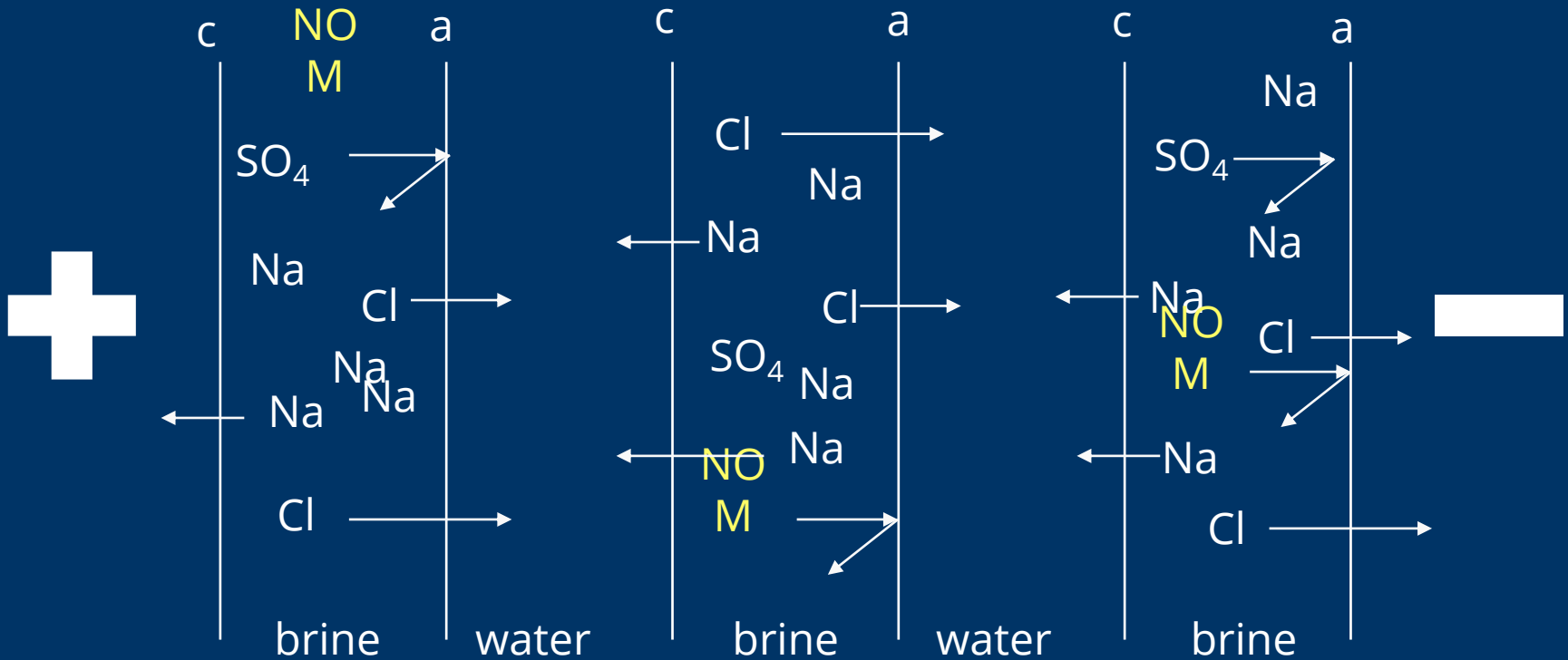
- stretch of C=O bond, indicates high polarity
  - higher than in most commercially available samples
- stretch of C=O in carbonyl linked to benzene ring
  - higher proportion of aromatic rings in fulvic acids
- presence of CH<sub>3</sub> and CH<sub>2</sub> in aliphatic parts of the structure, indicates hydrophobicity
- **HS from brine are more soluble and presumably have better chelating properties compared to other sources of commercial humic substances**

# separation of humic substances

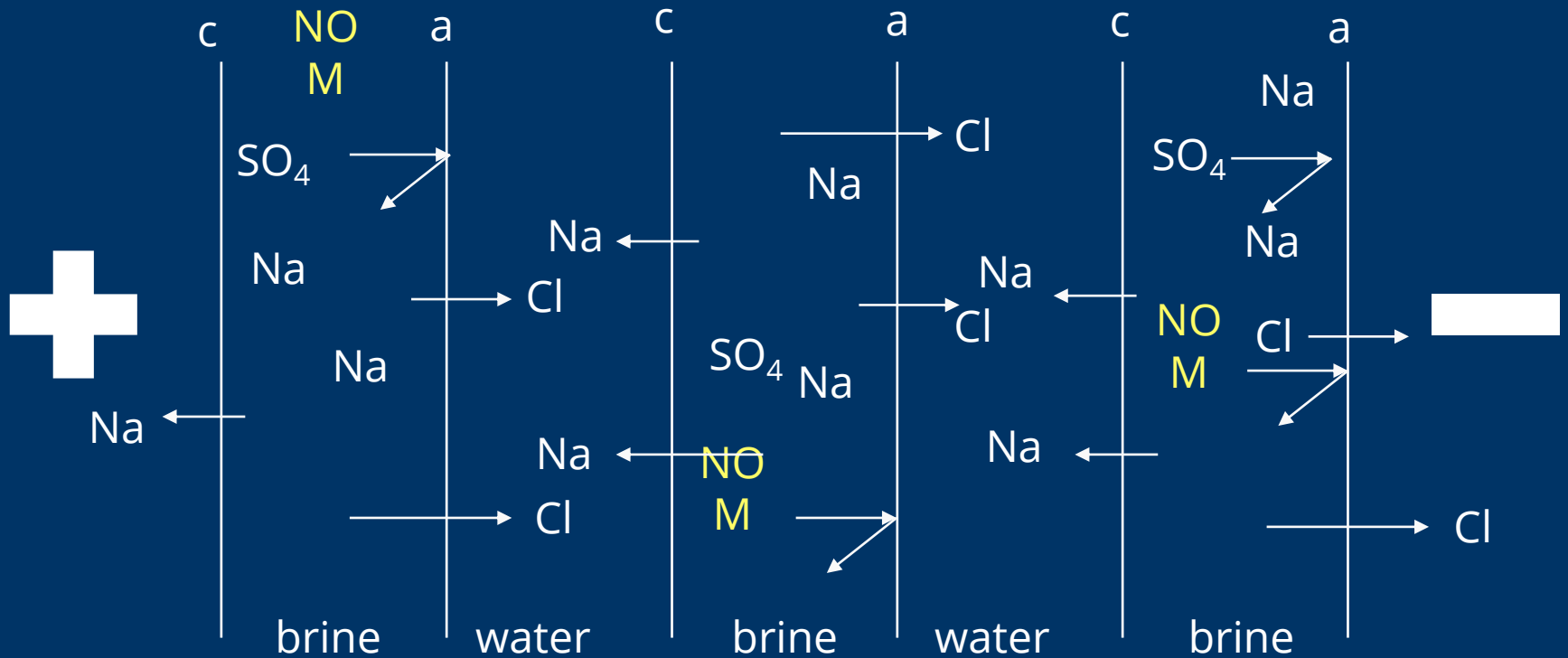
on industrial scale



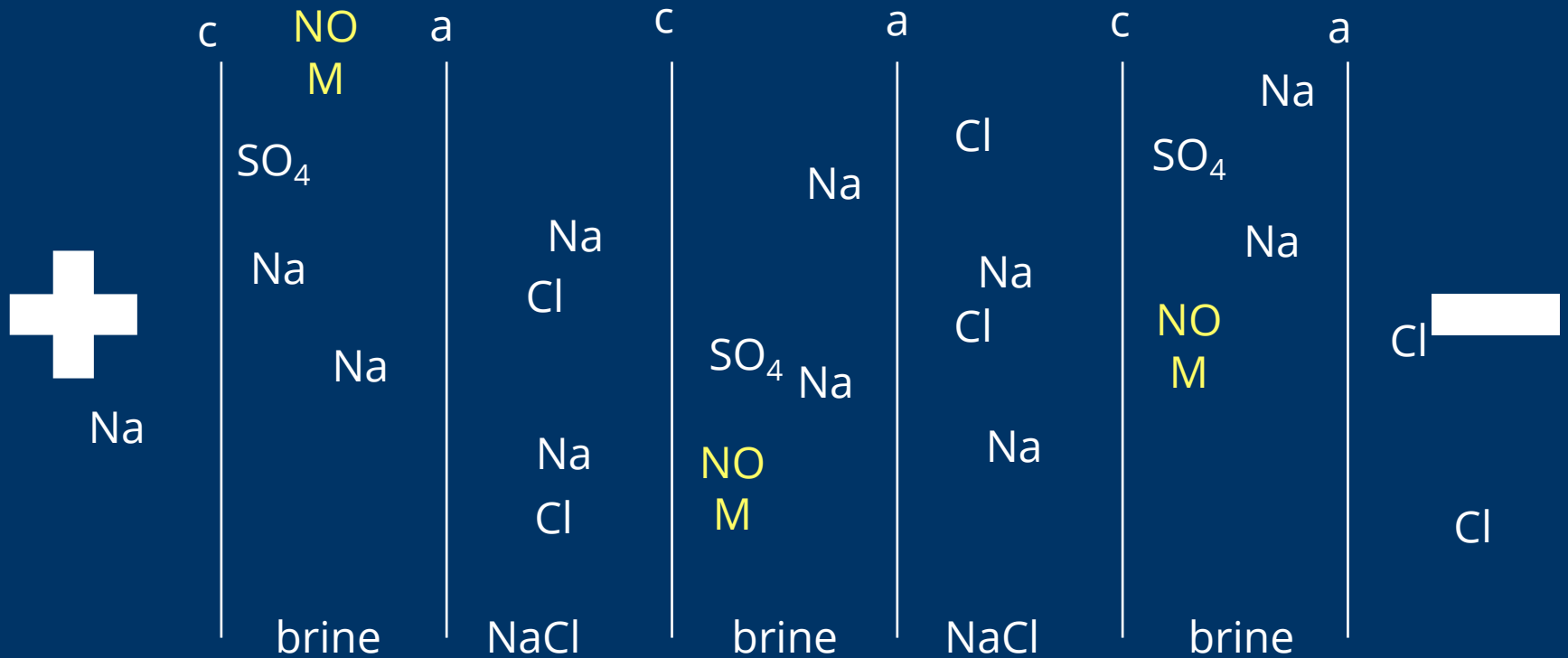
# mono-selective electro dialysis



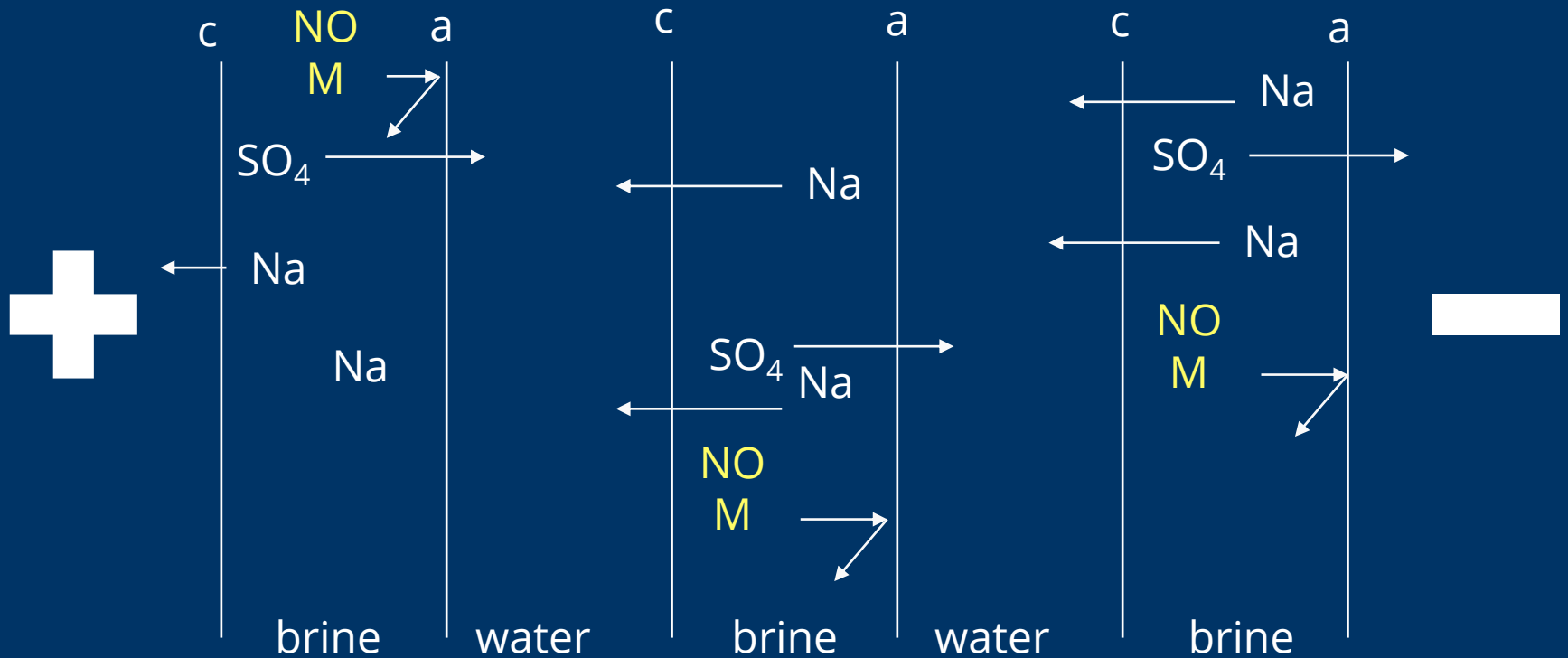
# mono-selective electro dialysis



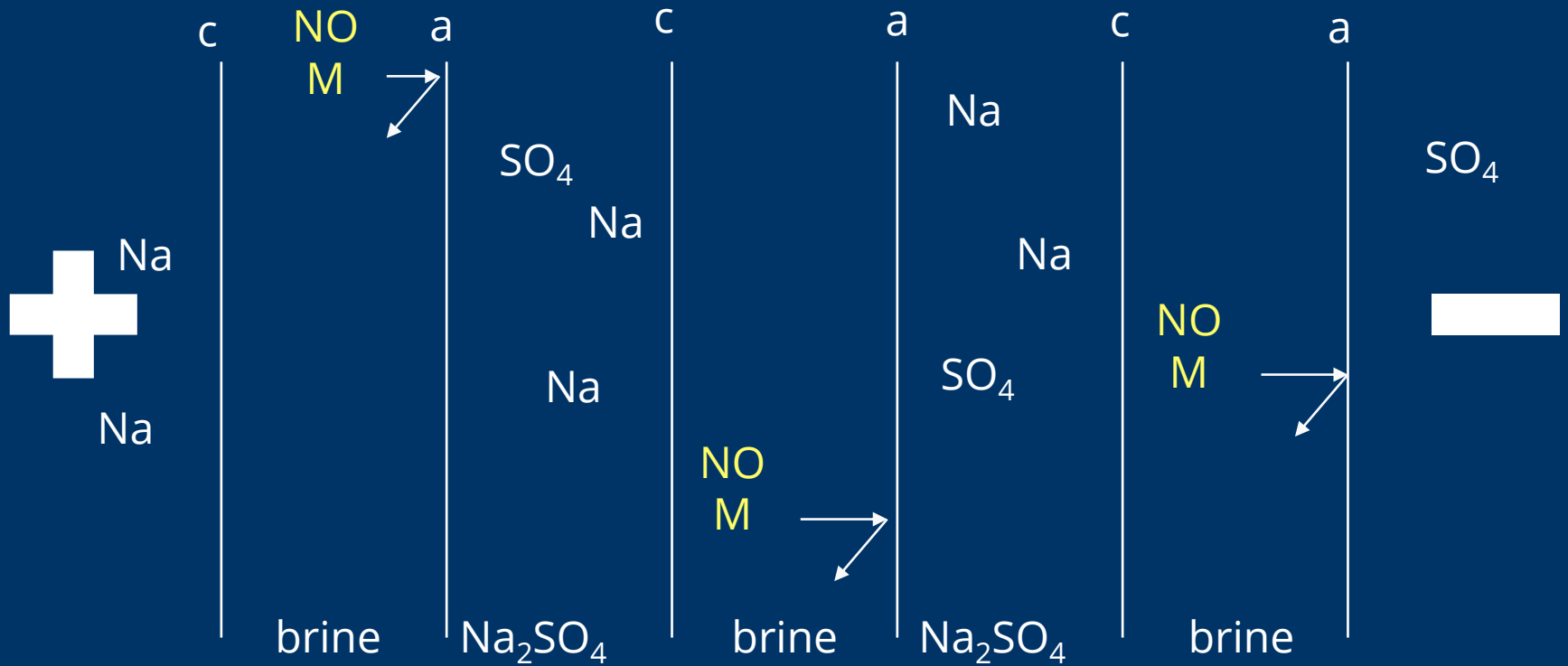
# mono-selective electro dialysis



# electrodialysis standard



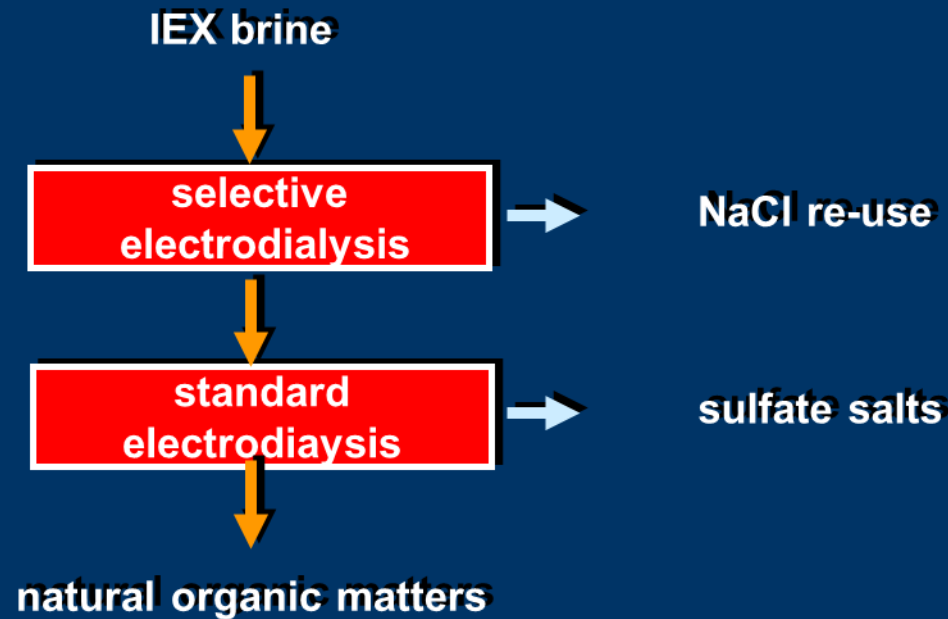
# electrodialysis standard





# separation of humic substances

on industrial scale



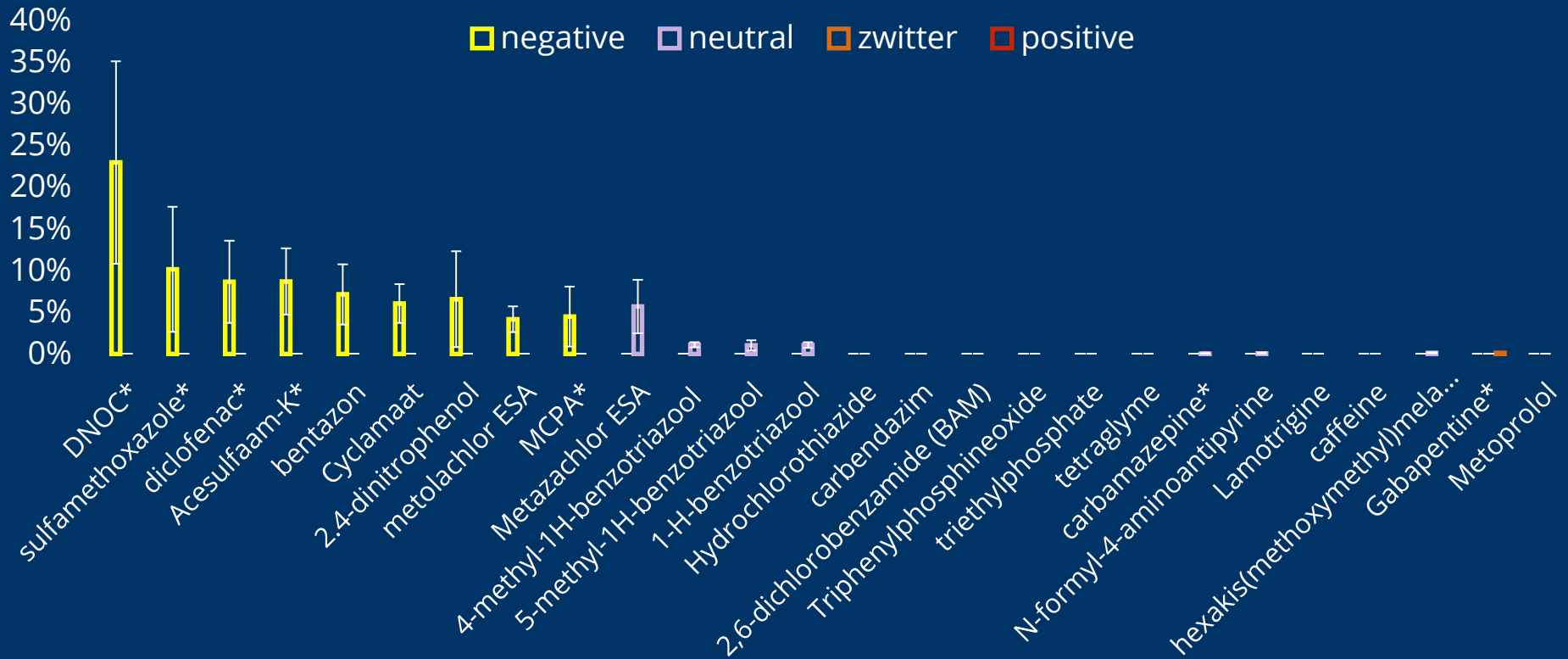
# contamination with organic micropollutants

## definition

- “organic substances whose toxic, persistent and bioaccumulative properties may have a negative effect on the environment and/or organisms. They are present in many products that we consume daily (drugs, cosmetics, phytosanitary products, insecticides, etc.), at the home or in industry.”

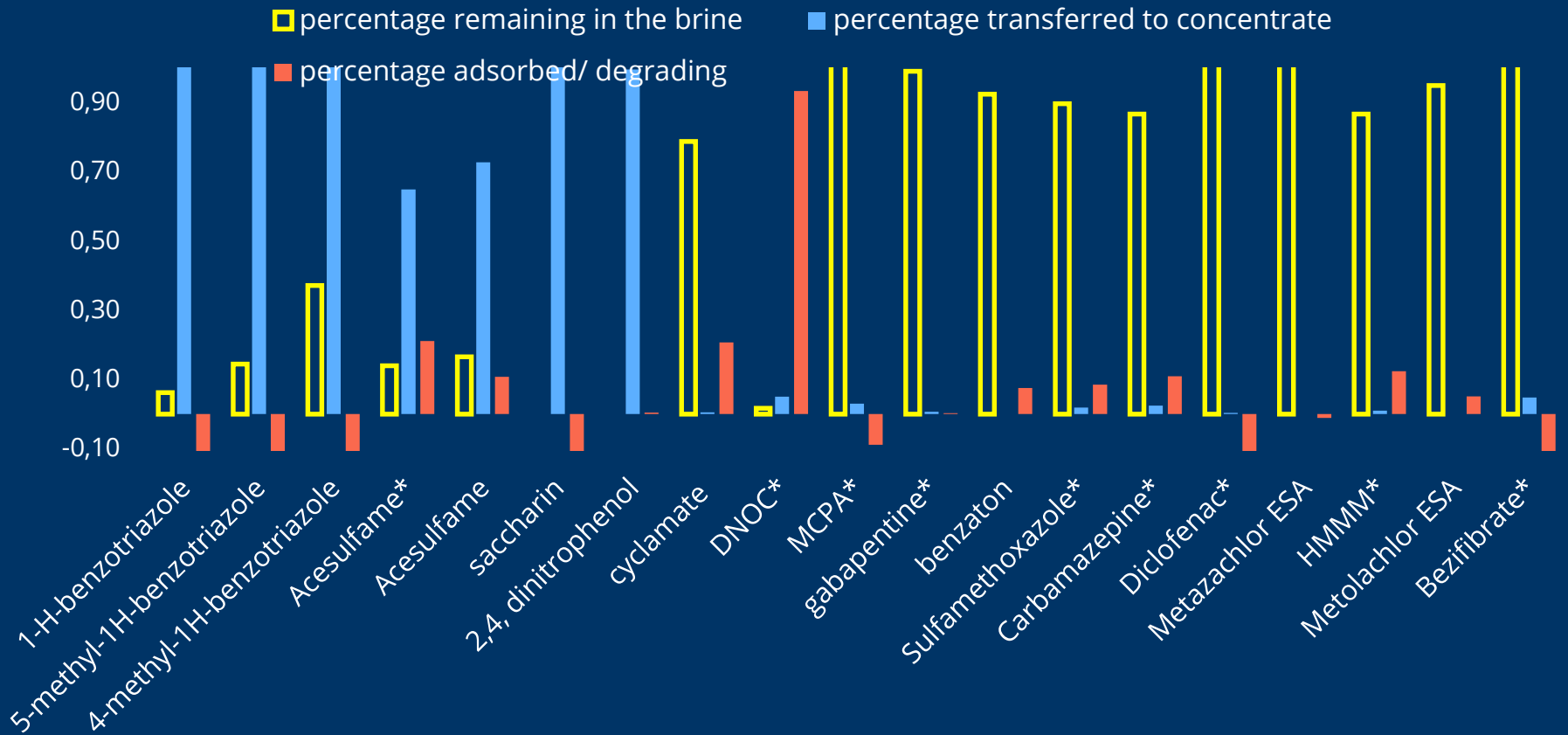
# contamination with micropollutants

micropollutants from IJssel lake concentrating in the brine



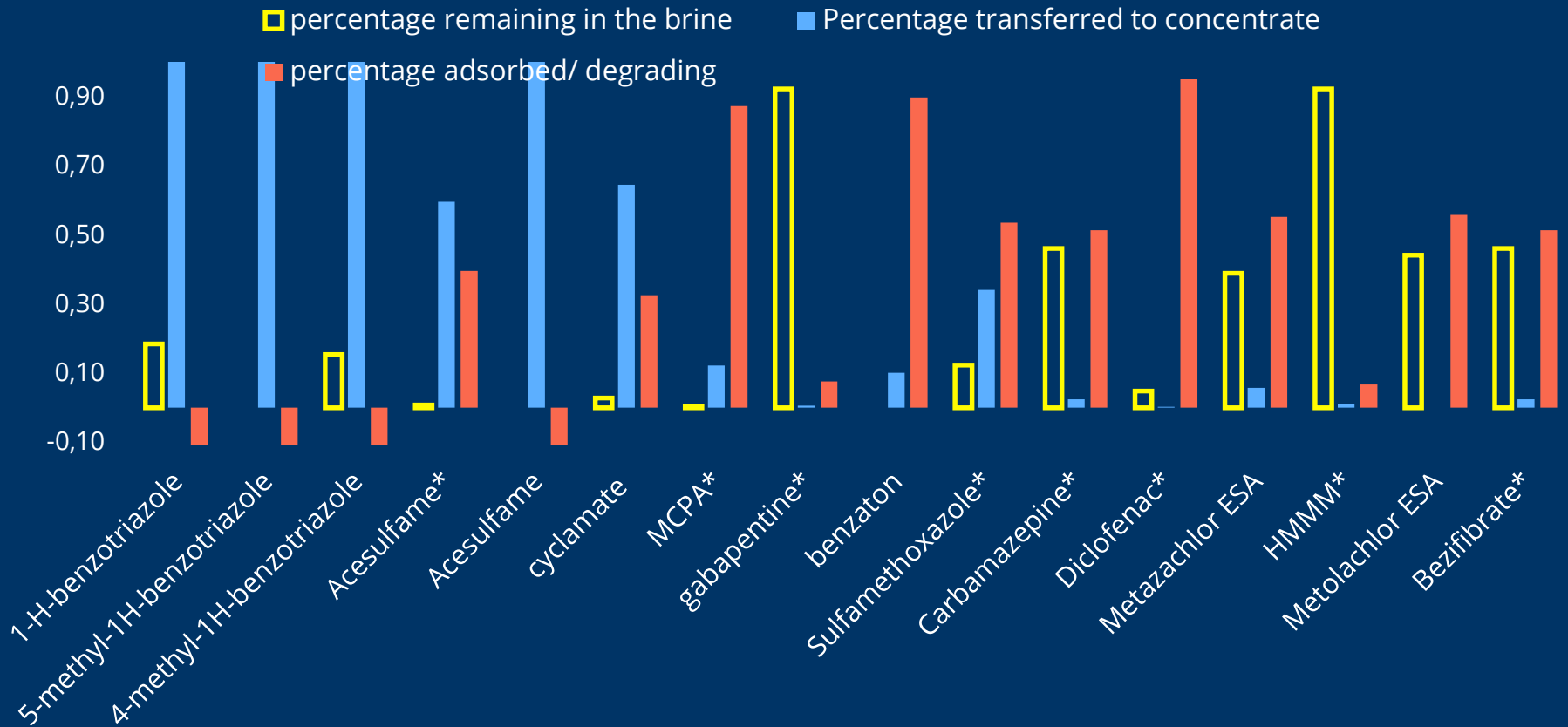
# contamination with micropollutants

passage from brine through mono selective electrodesialysis



# contamination with micropollutants

passage from ED effluent through electro dialysis standard



# contamination with micropollutants

## conclusions

- confirms the retention onto ion exchange resin of negatively charged organic micro-pollutants specifically
- explains the passage through selective and standard ion exchange membranes by van der waals radius:
  - smaller molecules only through selective membranes
  - most organic micropollutants species passing standard membranes
- no standards available for organic micro-pollutants limits in NOM

## applications

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Thank you!