

Solid-phase extraction of dissolved organic matter (SPE-DOM) from river, estuarine and open ocean waters

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Extraction methods

Solid-phase extraction (SPE)

using XAD resins

using sequential combination of different XAD resins

using various sorbents

using pre-packed cartridges and discs with silica-C18 sorbent

- Ultrafiltration with a 1 kDa cutoff
- Combination of ultrafiltration and SPE
- Sequential combination of reversed osmosis and electrodialysis

Properties of the solid phase sorbents

(according to manufacturer's (Varian) information)

Sorbent	Structure	Pore size	Retention properties
C18	octadecyl bonded phase, silica-based	60 Å	retention of non-polar compounds
C18EWP	octadecyl bonded phase, silica-based	500 Å	more efficient retention of large molecules, compared to C18
C18OH	non-endcaped octadecyl bonded phase, silica-based, with active silanol groups	150 Å	enhanced retention of basic compounds, compared to C18
C8	octyl bonded phase, silica-based	60 Å	not as retentive for non-polar compounds as C18
PPL	styrene divinyl benzene polymer	150 Å	retention of highly polar to non-polar substances from large volumes of water
ENV	styrene divinyl benzene polymer	450 Å	similar to PPL, larger pore size

Scheme for the isolation of SPE-DOM from seawater



DOM

* choose size of PPL cartridge according to sample volume and DOC concentration: do no exceed 2 mmol DOC or 10 L sample per g adsorber

Extraction procedure abord





PPL-Extracts



Extraction efficiencies for the isolation of SPE-DOM using different sorbents



North Brazil shelf and coastal zone



Composition of combined hydrolysable amino acids (North Sea)



Percentage of D-amino acids (North Sea)



Reversed-Phase High-Performance Liquid Chromatography (HPLC)

Comparison of PPL and C18 sorbent

Fluorescence and DAD detection



Fluorescence detector: ex: 260 nm; em: 430 nm DAD contour plot : 200-400 nm (700 nm)

All other parameters are identical, e.g. amount of enriched sample, elution volume, injection volume

Location of samples and DOC concentration



- A Apalachicola River and tributaries
- B Apalachicola salt marshes
- C North Brazil shelf and coastal zone
- D Gulf of Mexico deep sea
- E Weddell Sea (surface to bottom)





Extraction efficiency and C/N ratio of SPE-DOM (PPL)



- A Apalachicola River and tributaries
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Summary

Simple and robust method

PPL extracts >60% of coastal and >40% of deep-sea DOC

PPL retains a major fraction of N-containing compounds

Complete desalting of the sample NMR Ultra high resolution MS Various HPLC- and GC-MS-MS methods Fractionation of samples

Autonomous extraction with pumps from ship or on moorings







I urgently propose a workshop to decide on extraction methods for DOM

Thank you

Caeté Estuary (Brazil)







Concentration of total amino acids



Extraction efficiencies for the isolation of SPE-DOM

using different sorbents





Concentration of amino acids

