Analysis of estrogen-effective compounds in surface and wastewater samples by HPTLC-pYES

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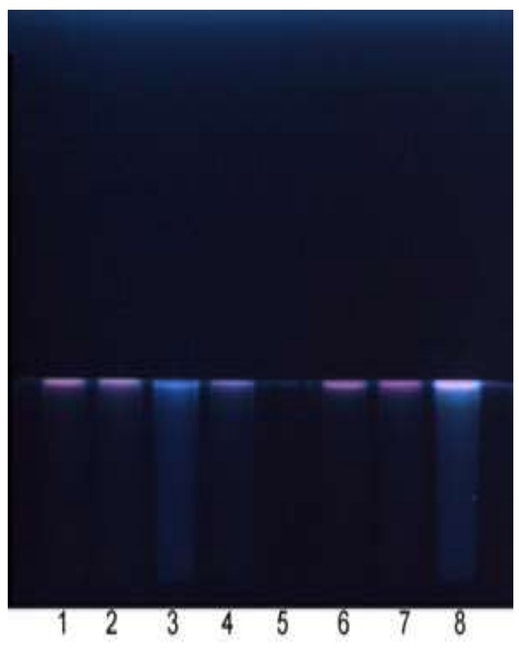
Highlights

- Liquid-liquid extraction was carried out with surface water and sewage treatment plant samples (untreated influent and effluent of primary clarifier)
- Qualitative detection of estrogen-effective compounds was performed by combination of HPTLC with the Yeast Estrogen Screen bioassay (pYES)
- Biodensitometry was used to determine the blue fluorescent dye 4-methylumbelliferone formed, indicating the estrogen-effective compound
- Targeted identification of the detected estrogen-effective compounds was performed by HPTLC-ESI-MS via the elution-head based TLC-MS Interface

APPLICATION

Rectangle 7 x 25 mm

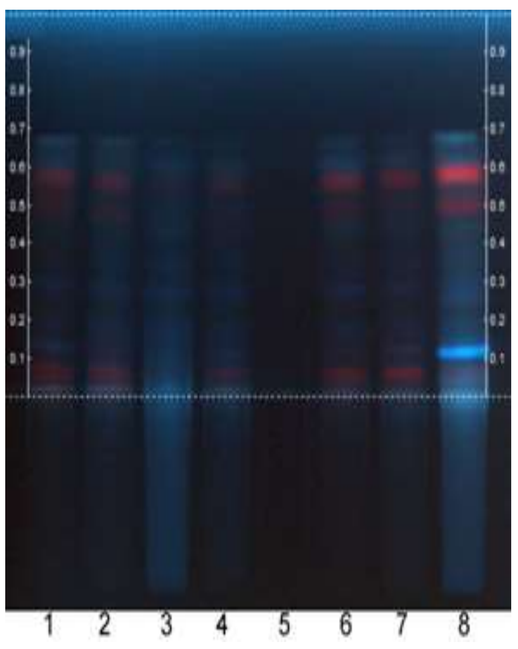
Dosage speed 800 nL/s



FOCUSING

Twofold front elution

1. methanol
2. ethyl acetate

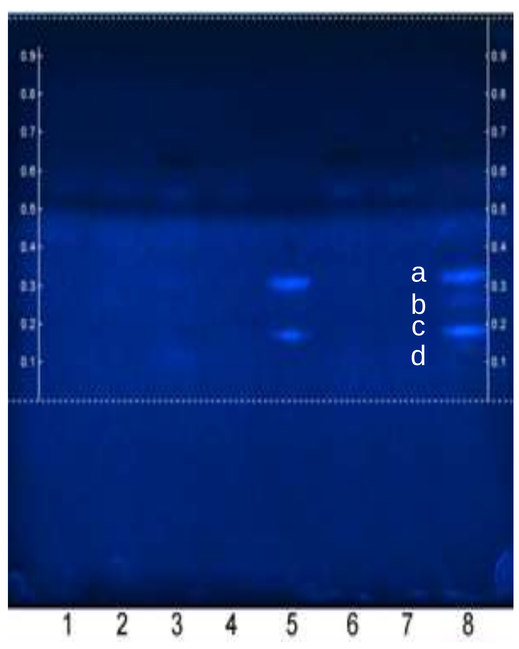


DEVELOPMENT

Solvent system consisting of

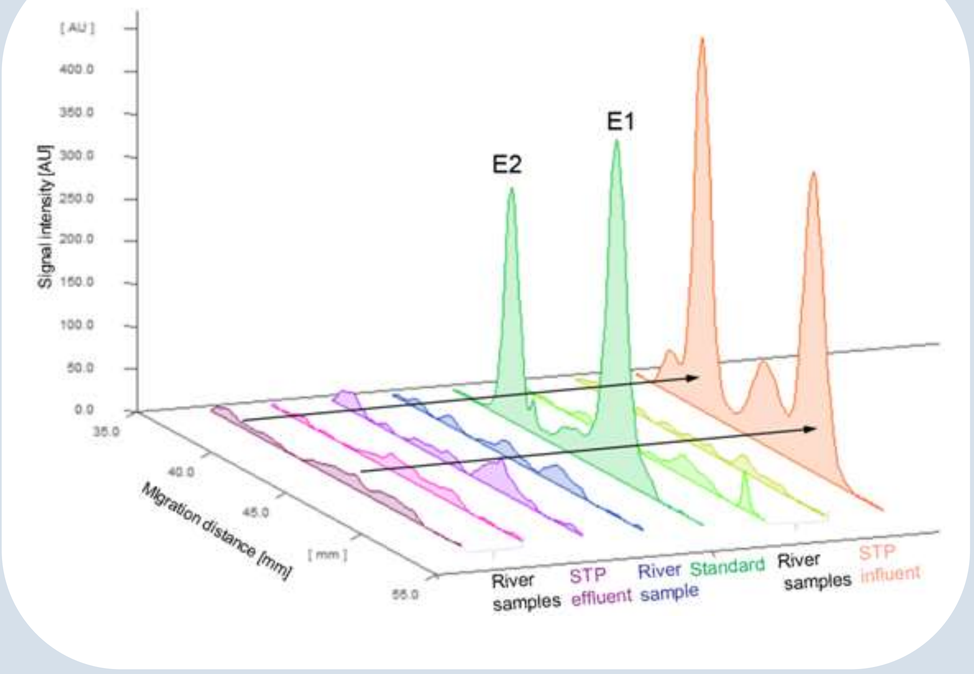
n-hexane - toluene - ethyl acetate

4:1.5:2 , *V*/*V*/*V*



BIOASSAY

Saccharomyces cerevisiae cells carrying the human estrogen receptor hERα



QUANTIFICATION

Fluorescence measurement at 366/>400 nm

IDENTIFICATION

Elution head-based TLC-MS Interface

Outcome

- Mean recovery rate of 88 % for 6 estrogen-effective compounds at this ultratrace level
- Non-target detection
- 17 samples analyzed in parallel
- Samples applied as native as possible: used directly or LLE
- Zone a identified as estrone (E1), zone c was 17β-estradiol (E2)
- Zones b and d are unknowns and need high-resolution MS recordings.
- In the influent of the sewage treatment plant, concentrations of E1 and E2 ranged from 3 to 50 ng/L, and for estriol (E3) from 98 to 210 ng/L. Ethinylestradiol (EE2), 4-*n*-nonylphenol (NP) and bisphenol A (BPA) were not detected.
- In every second surface water sample, E1 and E2 were detected, but not E3, EE2, BPA and NP. [1]

Literature [1] I. Klingelhöfer, G.E. Morlock, Anal. Chem. 87 (2015) 11098-11104

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