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EXTRACTION OF STEROID HORMONES FROM SLUDGE USING LIQUID MEMBRANE EXTRACTION TECHNIQUE

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INTRODUCTION

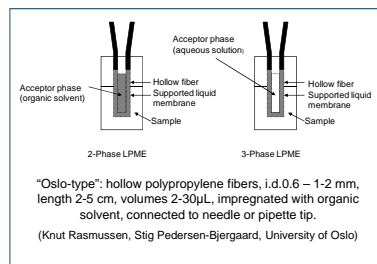
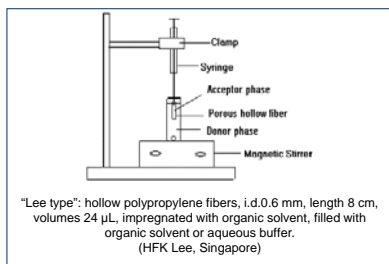
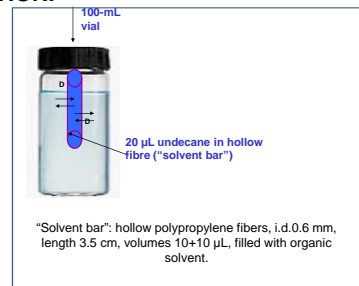
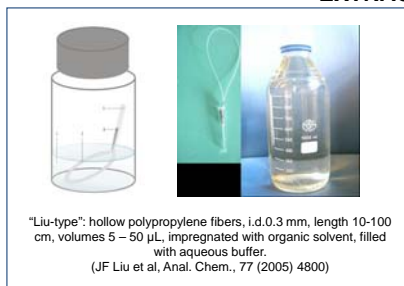
- Liquid Membrane Extraction Technique

- Liquid membrane extraction techniques permit the application of classical liquid-liquid extraction (LLE) chemistry to efficient and convenient operation. Various shortcomings of LLE are overcome by such extraction techniques as they use none or very little organic solvents, high enrichment factors can be obtained and there are no problems with emulsions.
- With a three phase SLM system (aq/org/aq), analytes are extracted from the aqueous sample into an organic liquid, immobilized in a porous hydrophobic membrane support, and further to a second aqueous phase. This is suitable for the extraction of polar compounds (acidic or basic, charged, metals, etc.) and it is compatible with reversed phase HPLC.
- A two-phase system (aq/org) where analytes are extracted into an organic solvent separated from the aqueous sample by a hydrophobic porous membrane is more suitable for hydrophobic analytes and is compatible with gas chromatography.
- The experimental format can be based on either flat membranes and on hollow fiber membranes in various ways.
- With other applications Membrane extraction techniques can be applied to solid matrices like sludge, particulate matter in air for extraction of analyte under consideration. In present study we are attempting to extract steroid hormones like Estrone, Estradiol, and Ethinylestradiol from sewage treatment plant sludge as an application of a liquid membrane extraction technique. Sample preparation and analysis will involve a hollow fiber two-phase membrane liquid-liquid extraction setup combined with gas chromatography mass spectrometry (GC-MS).

WHY LIQUID MEMBRANE EXTRACTION FOR SAMPLE PREPARATION?

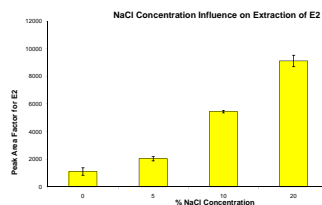
- Efficient clean-up (Better than Solid Phase Extraction)
- Nearly no organic solvents
- High enrichment factors
- No emulsion problems as in LLE
- Disposable, cheap and simple equipment
- Especially applicable to polar and semipolar compounds (acids, bases, zwitterionics, metals)
- Also for non-polar compounds (PCB, PAH, ...)
- Can be arranged with flat-sheet membranes or with hollow fibers
- With hollow fibers often called Liquid Phase Microextraction (LPME)
- Exhaustive extraction – gives total concentrations
- Equilibrium extraction – for determination of free fractions of pollutants

DIFFERENT HOLLOW-FIBER FORMATS FOR MEMBRANE EXTRACTION.

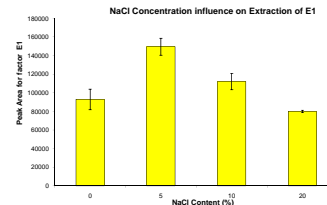


INITIAL FINDINGS

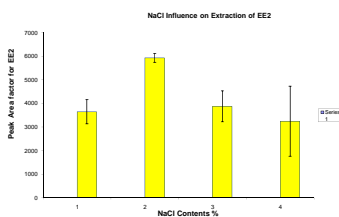
E1 Esterone



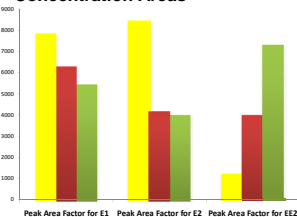
E2 Estradiol



EE2 Ethinyl Estradiol



Weight of Sludge Vs Hormones Concentration Areas



Recent membrane applications from the Lund group

Analytes	Samples	Enrichment, LOD	Phases	Reference
Drugs (Ibuprofen) and metabolites	Environmental water, incl. sewage	2000; 7 ngL ⁻¹	2	Zorita et al. J. Chromatogr. A 1157 (2007) 30
Alkyl phenols (incl. nonylphenol)	Environmental water	n/a (ESTM)	2	Liu et al. Anal. Chem. 78 (2006) 8526
Nitrophenols	Environmental water (incl. sea)	300; 0.1 μ g L ⁻¹	2	Bartolomé et al. J. Separation Sci. 30 (2007) 2144
PBDE	Environmental water, incl. leachate	5000, 0.2-1 ngL ⁻¹	2	Fontanals et al. J. Chromatogr. A 1133 (2006) 41
Steroid hormones	Environmental water, incl. sewage	1400; 1.5-10 ngL ⁻¹	2	Zorita et al. J. Chromatogr. A 1192 (2008) 1
Drugs (Salbutamol and Terbutaline)	Environmental waters, urine	50-200; 0.5-2.5 μ gL ⁻¹	3	Yamini et al. J. Chromatogr. A 1124 (2006) 57
Dinitrophenols	Environmental waters	300; 0.1 μ g L ⁻¹	3	Lezamiz et al. J. Chromatogr. A 1152 (2007) 226
Phenoxy acid herbicides	Environmental waters (very dirty)	400; 0.3 μ g L ⁻¹	3	Liu et al. J. Chromatogr. A 1160 (2007) 56
PhIP (aromatic heterocyclic amine)	Urine, plasma	126; 11 ng L ⁻¹	3	Lezamiz et al. Anal. Bioanal. Chem 390 (2008) 689
Drugs (fluoxetine, norfluoxetine)	Sewage	1700, 11 ng L ⁻¹	3	Zorita et al. J. Separation Sci. 30 (2007) 2513

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