Phenols with gradient LC-EC

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Introduction

Chlorophenols and nitrophenols are used in industry and agriculture for several purposes. In the end they may be found in river or drinking water. The MAC (maximum admissible concentration) in the EEC countries for phenols in drinking water is 0.5 µg/l. In the 70's the US environmental protection agency (EPA) created a list of the eleven most important phenol contaminants as priority pollutants.
In this application an HPLC method for the analysis of the 11 EPA phenols in water is described using electrochemical detection. Detection limits are between 0.1 and 2 ppb.

Method

A linear gradient running from 25 to 45% ACN in 20 min appeared to be favourable (Fig. 1).
The detection limit of the phenols is strongly related to the injection volume and the sample pre-treatment that is used. In principle, a 100 fold larger sample volume will result in a 100 fold better detection sensitivity (Fig. 2). A pre-requisite is that the solvent front, system peaks and possible contaminants do not interfere with the analysis.

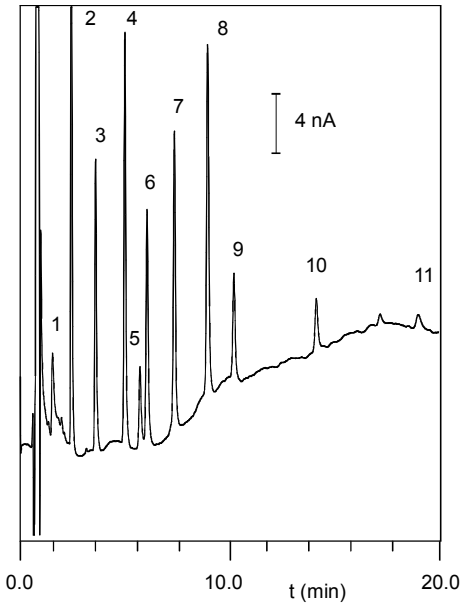

Fig. 1. Analysis of a standard mixture of 200 ppb phenols: 1: 2,4-dinitrophenol (DNP), 2: phenol (P), 3: 4-nitrophenol (4-NP), 4: 2-chlorophenol (2-CP), 5: 2-methyl-4,6-dinitrophenol (MDNP), 6: 2-nitrophenol (2-NP), 7: 2,4-dimethylphenol (DMP), 8: 4-chloro-3-methylphenol (CMP), 9: 2,4-dichlorophenol (DCP), 10: 2,4,6-trichloro-phenol (TCP), and 11: pentachloro-phenol (PCP).

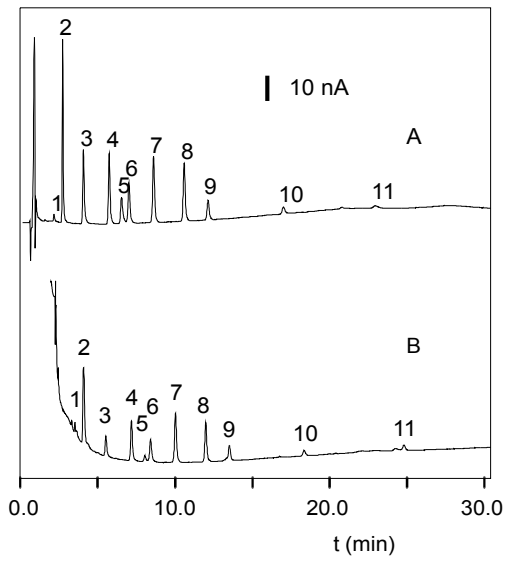

Fig. 2. Improvement in determination limit by large volume sample injection. Injection volume and concentrations for each phenol are A: 20 µl (200 ppb) and B: 2 ml (2 ppb).

References

1. J. Ruana, I. Urbe, F. Borrull, Determination of Phenols at the ng/l Level in Drinking and River Waters by Liquid Chromatography with UV and Electrochemical Detection, J. Chromatogr. A 655 (2) (1993) 217-226

LC-EC conditions

Column	Spherisorb ODS2, 100x4.6 mm, 3 µm
Flow rate	1.5 ml/min
Mobile phase	50 mM HAc/NaAc, pH 4.0, 25-45% ACN gradient
Temperature	30 °C
E-cell	1200 mV (vs. Ag/AgCl sat'd)

Part numbers and configuration used

120.0035	DECADE EC detector
110.4105	VT-03 flow cell with 3mm glassy carbon WE and salt bridge Ag/AgCl REF

Configuration suggestions & comment

See application note 216-001.