PHENOLS IN WATER

THE BRIGHTEST LC-EC APPLICATIONS FOR ENVIRONMENTAL ANALYSIS EVER PLOTTED OUT

Chloro- and nitrophenols

- *2,4-dinitrophenol (DNP)*
- *phenol (P)*
- *4-nitrophenol (4-NP)*
- *2-methyl-4,6-dinitrophenol (MDNP)*
- *2-chlorophenol (2-CP)*
- *2-nitrophenol (2-NP)*
- *2,4-dimethylphenol (DMP)*
- *4-chloro-3-methylphenol (CMP)*
- *2,4-dichlorophenol (DCP)*
- *2,4,6-trichlorophenol (TCP)*
- *pentachlorophenol (PCP)*

Ketones

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) made a list of the eleven most important phenol contaminants as priority pollutants. The standard EPA method is based on a concentrating liquid-liquid extraction followed by derivatisation and GC analysis with electron capture detection.

In this application a HPLC method for the analysis of the 11 EPA phenols in water is described using electrochemical detection. Detection limits are between 25 and 220 ng/l, except for DNP (0.9 µg/l), TCP (0.95 µg/l) and PCP (6 µg/ml).

- Chlorophenols in industry and agriculture.
- Nitrophenols in industry and agriculture
- 11 EPA phenols in water

Summary

The ALEXYS Phenols Analyser is routinely applied for analysis of environmental phenols. Phenols are analysed routinely using an isocratic or gradient LC system. Detection limits in the low PPB range are obtained. By using on-column sample concentration in combination with large volume in jection the detection limits are easily improved by a factor 10 – 100.

Fig 1. ALEXYS Phenols Analyzer.

Method

The detection potential is optimised by constructing I/E relationships for 9 phenols. Due to the poor detection characteristics of some phenols, especially 2,4 DNP, a working potential of 1200 mV vs. Ag/AgCl is used for further experiments. The background current is approximately 200 nA.
Working electrode contamination was only problematic at high phenol concentrations. The concentrations in samples are in the low ppb range or lower. At the ppb level no significant contamination of the working electrode could be measured within one day. One cleaning procedure at the end of each day is sufficient to maintain reproducible working conditions.

Table 1

LC-EC conditions	
HPLC	ALEXYS Phenols Analyzer
Sample	100 - 1000 nM phenols, 20 µl injection
Temperature	30 °C

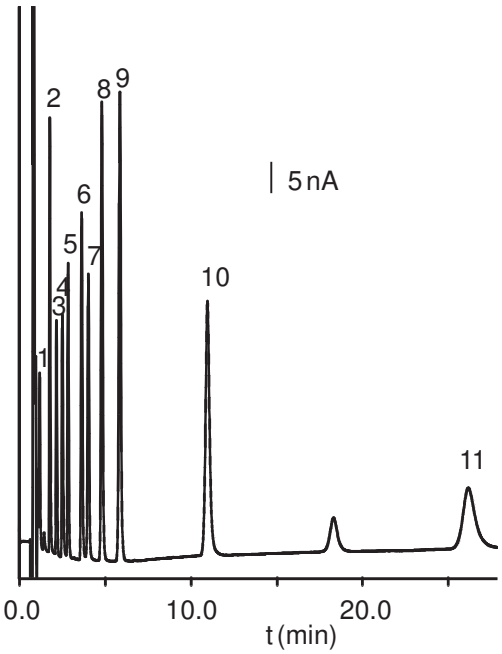

Fig. 2. Analysis of a standard mixture of phenol.

Concentrations in Fig. 1 (in ppb): 1: 2,4-dinitrophenol (DNP, 1840), 2: phenol (P, 90), 3: 4-nitrophenol (4-NP, 140), 4: 2-methyl-4,6-dinitrophenol (MDNP, 400), 5: 2-chlorophenol (2-CP, 130), 6: 2-nitrophenol (2-NP, 280), 7: 2,4-dimethylphenol (DMP, 120), 8: 4-chloro-3-methylphenol (CMP, 290), 9: 2,4-dichlorophenol (DCP, 820), 10: 2,4,6-trichlorophenol (TCP, 1970), and 11: pentachlorophenol (PCP, 2660).

A linear gradient running from 25 to 45% ACN in 20 min appeared to be favourable (Fig. 3).
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. A pre-requisite is that the solvent front, system peaks and possible contaminants do not interfere with the analysis.

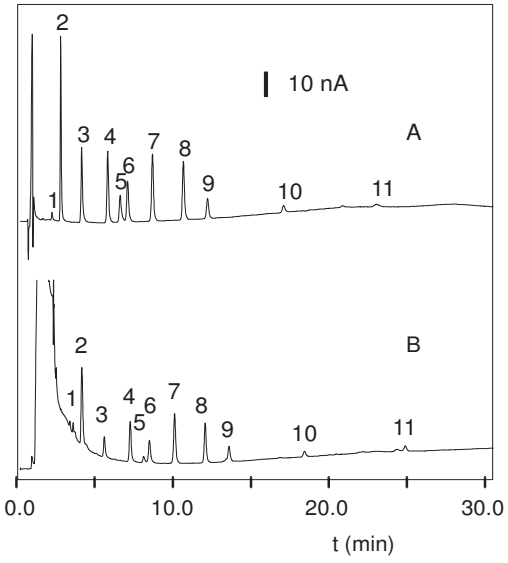

Fig. 3. Improvement in determination limit by large volume sample injection. Injection volume and concentrations for each phenol are A: 20 µl (200 ppb) and B: 2 ml (2 ppb).

Conclusion

An ALEXYS LC-EC system has been used for the analysis of phenols in water. Gradient HPLC is most suitable for analysing multiple phenols.

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

PART NUMBERS AND CONFIGURATIONS	
180.0094A	ALEXYS Phenols Analyzer