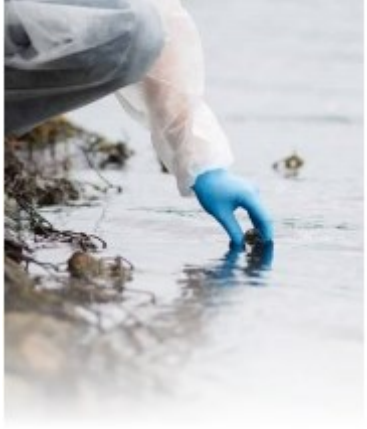
Application Note
Environmental



Phenols in Water

The finest LC-EC Applications for Environmental analysis

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)
PET Ketones

- **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 injection the detection limits are easily improved by a factor 10 – 100.

In this application a HPLC method for the analysis of the 11 EPA phenols in water is described using electro-chemical 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).

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.

Figure 1: ALEXYS Phenols Analyzer

Table 1

Conditions	
HPLC	ALEXYS LC-EC Analyzer
Column	Spherisorb ODS2, 100x4.6 mm, 3 µm
Mobile phase	50 mM HAc/NaAc, pH 4.0, 35% acetonitrile
Flow rate	1.5 mL/min
Injection	20 µL (full loop)
Temperature	30°C for separation & detection
Flow cell	SenCell* with GC WE, HyREF, AST 2
Potential	1.0 V vs HyREF
I-cell	about 200 nA
ADF	0.5 Hz
Range	100 nA/V

*Original work with VT03 cell 3mm GC vs sb REF.

Experimental

A DECADE II electrochemical detector (Antec Leyden, The Netherlands) equipped with a VT03 electrochemical flow cell (Antec Leyden) with 2 mm diameter glassy carbon electrode and 25 µm spacer was used for all experiments. Effective cell volume determined by electrode area and spacer thickness, was 71 nl.

Results

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.

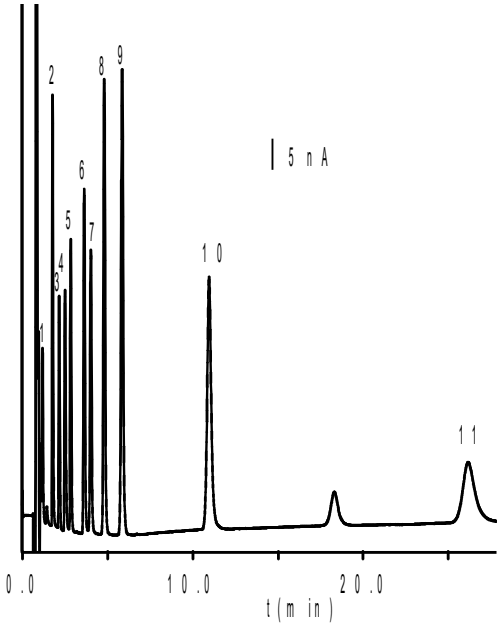


Figure 2: Analysis of a standard mixture of phenol.

Concentrations in Fig. 2 (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. I n principle, a 100 fold larger sample volume will result in a100 fold better detection sensitivity. A pre-requisite is that the solvent front, system peaks and possible contaminants do not interfere with the analysis.

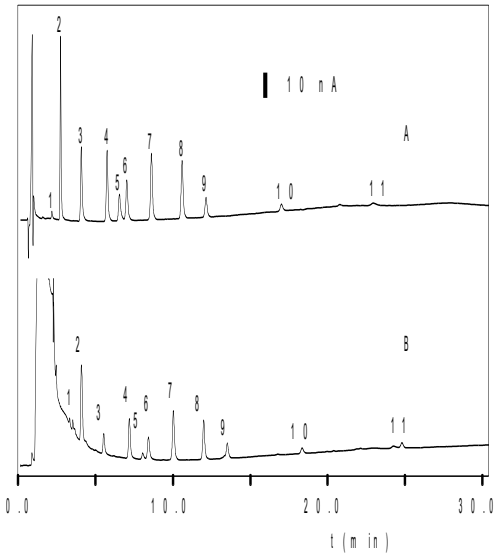


Figure 3: Improvement in determination limit by large volume sample injection. Injection volume and concentrations for each phenol are A: 20 µL (200 ppb) and B: 2 mL (2 ppb).

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

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.

Ordering information

180.0094E	ALEXYS Phenols Analyzer

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