

'...there is little fluctuation of the system between injections, achieving good reproducibility...'

Determination of Phenols by Electrochemical Detection

Abstract

A method is described for the analysis of phenols in waste water by RP-LCEC (Reverse Phase Liquid Chromatography with Electrochemical Detection). The method is highly selective while no derivatisation is required. Sensitivity is at ppb level. There is little fluctuation of the system between injections, achieving good reproducibility and allowing samples to be analyzed every 15 minutes.

Phenols constitute a large part of the environmental pollutant load of many industrial processes, e.g., oil refining, coal processing, agrochemical manufacturing and wool preserving. Phenols are also found in soil from contaminated sites either as a direct result of past industrial practices or as degradation products.¹

The once popular colorimetric method of 4-aminoantipyrine derivatisation² has been replaced by modern instrumental methods. Gas chromatography employing mass spectrometry, electron capture and flame ionization detection is used but usually requires derivatisation of the phenols to improve volatility and sensitivity.³ By comparison, LCEC offers very high sensitivity (at pg level) and selectivity, in that detected compounds must be electroactive at the control potential. In addition, only minimal sample clean-up is required in most cases. Phenols, with the electroactive aromatic moiety, have been well studied in their trace analysis for environmental monitoring by LCEC. In fact, reverse phase LCEC has proven to be the most selective and sensitive of all modern techniques.

The optimum oxidation potential range for phenols is 700 to 1100 mV. Passivation of the glassy carbon electrode surface by the phenoxy radical, the oxidation product of phenol, is well documented. As a result of this passivation, the phenol oxidation potential is usually set at the upper limit of the available potential window,

Keywords:
Phenols, Electrochemical, RP-LCEC, Wastewater, Environmental

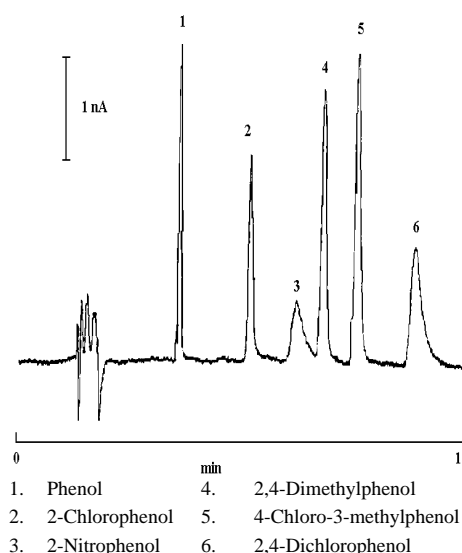


Figure 1 HPLC Separation of Various Phenols

typically at 1110 mV. The optimum oxidation potential for substituted phenols tends to be less, depending upon the substituent's ability to stabilize the aromatic nucleus.

The GBC LC1260 Electrochemical Detector, with its unique 'Wall Jet' design, allows shorter equilibration time and increased reliability. During normal operation, it is believed that the high turbulence created in the 'Wall Jet' nozzle decreases chemical fouling of the working electrode surface, thereby reducing the requirement for the detector flow-cell to be dismantled frequently for cleaning. Sensitivity is enhanced through the use of low noise electronic circuitry with active and digital filtering.



E1

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E1
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Conditions

Column: Spherisorb S5 ODS2,
150 x 4.6 mm ID
Mobile Phase: 0.02 M Ammonium Acetate,
pH 6.0/Acetonitrile (60:40)
(Helium Sparging)
Flow Rate: 1.0 ml/min
Temperature: Ambient
Detection:
Working Electrode: 3 mm
MicroComposite
Glassy Carbon
Reference Electrode: Ag/AgCl (3M KCl)
Auxiliary Electrode: Cell Body
Applied Potential: 1100 mV

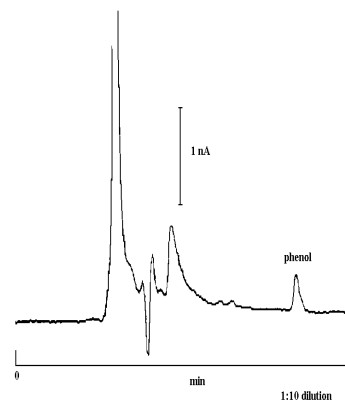


Figure 2

Sample Preparation

1. Dilute sample with mobile phase.
2. Filter diluted sample through a 0.2 µm filter.
3. Transfer small quantity into autosampler vial.

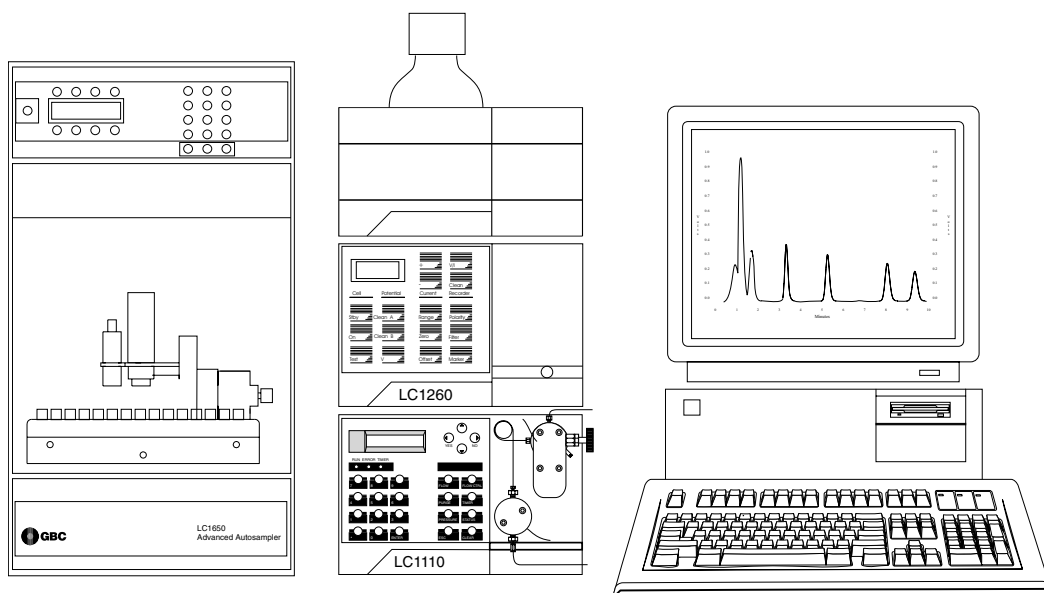
(As with all preparation procedures, the analyst must determine the suitability or otherwise of the above procedure for their sample type. This procedure is for relatively clean wastewater, free of suspended matter.)

GBC HPLC Instrumentation

LC1110 Dual Piston HPLC Pump
LC1260 Electrochemical Detector
LC1440 System Organiser
LC1650 Advanced Autosampler
WinChrom Chromatography Data
Management System

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