

Characterization of Organic Pollutants in Industrial Effluents Using Liquid Chromatography–Atmospheric Pressure Chemical Ionization–Mass Spectrometry

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Contaminated industrial effluents often contain a variety of organic pollutants which are difficult to analyse by standard GC–MS methods since this technique often misses the more polar or non-volatile fraction of these organic compounds. In the present work a method for the characterization of complex mixtures of organic contaminants present in various industrial effluents is proposed. The protocol consists of setting-up a methodology based on solid phase extraction (SPE) using an Automated Sample Preparation with Extraction Columns system (ASPEC XL) and Lichrolut EN sorbent material for preconcentrating 300–500 ml of water volumes spiked with a variety of pollutants: phenolic compounds, benzophenone, isothiocyanate-cyclohexane, ethylbenzoate, 1-methyl-2-pyrrolidinone, 2-methylbenzenesulphonamide, benzidines, acridine, 1,1,3,3-tetramethyl-2-thiourea, 2,2-dimethyl-1,3-propanediol, phosphates, phthalates and non-ionic detergents characterized by LC–MS using atmospheric pressure chemical ionization in the positive and negative ion modes. The developed protocol permitted unequivocal identification of target analytes such as pentachlorophenol, tributyl phosphate, 4-nonylphenol, dibutylphthalate, dimethylphthalate, bis(2-ethylhexyl)phthalate, isothiocyanate-cyclohexane, ethylbenzoate, 2-methylbenzenesulphonamide, tetramethyl-thiourea, 2,2-dimethyl-1,3-propanediol and 1-methyl-2-pyrrolidinone at concentration levels varying from 0.16 to 54.4 $\mu\text{g l}^{-1}$. © 1997 by John Wiley & Sons, Ltd.

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INTRODUCTION

A great number of synthetic organic chemicals have been released to the environment. Many of them are toxic at low concentrations or may accumulate in sediments and organisms. As a consequence, strict characterization of contaminated effluents needs to be done. In this respect the European Union (EU) promulgated several years ago a so-called 'blacklist' of 132 dangerous substances (Directive 76/464/CEE) of target analytes that should be monitored as dangerous substances discharged into the aquatic environment.¹ The blacklist includes several organohalogenes such as poly-

chlorinated biphenyls (PCBs), chlorotoluenes and chloropropanes, some organophosphorus compounds such as pesticides and tributylphosphate, chlorophenols and polycyclic aromatic hydrocarbons (PAHs). Recently a new Directive on Integrated Pollution Prevention Control (IPPC) has been promulgated by the EU.² This new Directive expands the range of pollutants that should be monitored in industrial effluent discharges and involves a multiannual work programme that covers many industrial sectors such as the paper and pulp industry, refineries and textiles. It is indicated in the Directive that all the substances discharged by the various industrial sectors should be monitored and the former EU blacklist expanded by adding new compounds. It is of interest to the EU to develop monitoring strategies for the characterization of a variety of pollutants. From this perspective, research in the area of characterizing new pollutants in contaminated industrial effluents will be encouraged and expanded during the coming years.

Common methods for identifying organic pollutants in contaminated industrial effluents generally involve

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