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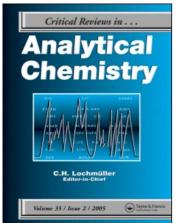
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TRACE ANALYSIS OF SOME ORGANIC COMPOUNDS IN ENVIRONMENTAL SAMPLES BY HPLC.

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The trends in environmental detection limits during last 30 years increases towards sensitivity and lower detection limit. It is consistent with changes in technology, both in the measurement and health sciences. In each decade, the trend has been the same, working toward lower detection limits and with increasingly complex matrices. Today many laboratories routinely use methods working at the parts per trillion (ppt) level. It is reasonable to expect that analyses will be targeted at ppt level in the 1990s and there is no fundamental reason why this trend of the last 30 years should change.

To achieve higher sensitivities, the determination will need new or enhanced detection technologies and will need to put more emphasis on sample preparation, matrix clean-up techniques, and preparation techniques. A number of new techniques have become available such as solid phase extraction, supercritical fluid extraction, membrane extraction, electrochromatography and solute focusing techniques.

The use of HPLC has some advantage:

- wide variety of selective interaction in LC
- availability of number of very selective devices (ampermetric, fluorometric, diode

array ...) that uniquely detect certain components at very low concentrations

• less sample cleanup required prior to LC analysis then in other method (for example GC); in favourable cases, samples can be injected without prior treatment or sample derivatization.

LC analysis (sensitivity, accuracy and reproducibility) depend on:

- column resolution
- chromatographic sample injection technique
- detection
- treatment of the sample

Improving of the detectability of a sample component:

- mass overload on a pre-column followed by either isocratic or gradient development
- post or pre-column derivatization using a detector sensitive to the attached chromophore
- membrane enrichment
- super critical fluid extraction

This phenomenon was used for trace analysis of some toxic organic compounds.

Concern about environmental pollution and occupational hazards due to the presence of carcinogenic compounds in air or water has led to the development of analytical methods for the determination at trace level. HPLC is a powerful and widely used technique for the analysis of trace components. There are several reasons why HPLC method is often used for the determination of toxic organic compounds in the environment: there is the wide variety of selective interaction; the high resolving power needed for trace analysis in complex mixtures; the availability of a number of very selective detectors (ampermetric, fluorometric, photothermal, two-photon excited fluorescence detection, DAD), that detect certain components at very low concentrations; there is often less sample cleanup required prior to LC analysis than in other methods and various preconcentration technique can be used in on line system. Each technique requires some special instrumental modification, although no system can be characterised as too

difficult or costly to assemble. Some examples of trace analyses of toxic compound in environmental will be given.

A method was proposed for the determination aliphatic aldehydes in river and tap water. This method utilised the separation of the aldehydes as their 2,4- dinitrophenylhydrazones derivatives by HPLC using on line preconcentration and gradient solvent elution. The reaction of the derivatization was studied on a microscale in water solution at different pH. It was found that aldehydes in spiked sample (at 1 ppb level) are decomposed and unknown compounds were formed. The limits of the detection at a wavelength of 355 nm and a signal/ noise ratio of 5 range from 50 ppt for formaldehyde to 200 ppt for butyraldehyde. The mean relative standard deviations for all aldehydes were 10 % at 1 ppb level [1].

A simple and rapid method for the determination of ethanolamine in water was developed [2]. The problem of ethanolamine preconcentration was solved by adding octane- 1 - sulphonic acid (OSA) to the water sample and ion pair was sorbed on the precolumn C18. After flushing ion pair of ethanolamine was desorbed to the analytical column with lower concentration of OSA in mobile phase. The retention of ethanolamine in ion-pairing reversed phase chromatography was modelled via changing the volume percentage of methanol and concentration of ion-pairing agent octylsulfonic acid in the mobile phase. Electrochemical detection was used. The limit of the detection at 0.88 V and signal/ noise of 5 was about 2 ppb.

A mathematical procedure which utilises the individual spectra of fifteen polyaromatic hydrocarbons was suggested for the optimisation of the detection wavelength [3]. Procedure is based upon use of weighting factors appropriate for the components under consideration (toxicological data). Two criteria have been used in the optimisation procedure. If suggested criterion was used and all the components were of equal importance, then the optimum was at 220 nm and 254 nm respectively. When the weighting factors based on the toxicological data were employed, the optimum was found at 287 nm.

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