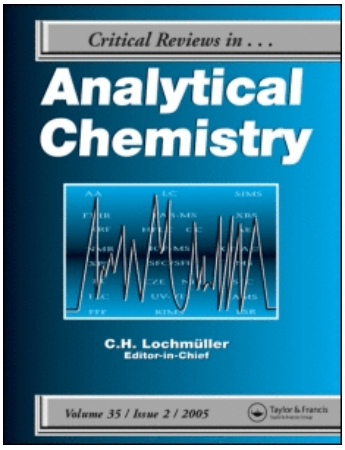

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ON THE CAPILLARY GAS CHROMATOGRAPHIC DETERMINATION OF PETROLEUM HYDROCARBONS IN ENVIRONMENTAL SAMPLES

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The identification and quantification of organic pollutants in environmental matrices are necessary for solving various environmental problems. Contamination of soils by petroleum hydrocarbons poses a major problem. Causes include crude oil leaks from tankers or pipelines, spills around production areas, disposal of refinery wastes, and transport fuels leaking underground storage tanks or railroad cars [1-3]. Petroleum hydrocarbons are extremely complex class of compounds with a wide range of physical and chemical properties. They are usually grouped as distillate fractions according to their carbon atom number and boiling point ranges, such as gasolines (C_3-C_{10}, 42-175°C), kerosene (C_{10}-C_{13}, 175-240°C), diesel (C_{13}-C_{20}, 240-340°C), light gas oil (C_{20}-C_{30}, 340-450°C), heavy gas oil (C_{30}-C_{44}, 450-538°C), and non-distillates (CAN>C_{44}, BP>538°C). Contamination of soils with petroleum hydrocarbons can involve some or all these fractions.

Contamination of soil with petroleum hydrocarbons has become a major environmental concern and has driven to the development of analytical procedures both for detection of threats to underground quality, such as underground fuel tank or landfill leakage, as well as characterisation of the real extent and magnitude of existing contamination to effectively assess risk and design remedial actions.

The typical procedure for determination of petroleum hydrocarbon pollutants in contaminated soil usually consists of [1-3]:

- sampling the soil samples from contaminated soils,
- extraction of hydrocarbons from soil samples by a gas, supercritical fluids, or an organic solvent,
- HRGC analysis of hydrocarbons in extracts.

An alternative method for the determination of pollutants in contaminated soils is based on the analysis of interstitial subsurface pore gases for indication of underlying or nearby contamination by volatile [4]. In this method interstitial subsurface pore gases (soil gases) are sampled and analysed for detection of underlying or nearby contamination by volatile organic compounds . Method consists of:

(1) Sampling which can be performed through:

a) Collection of soil gases in metal canisters, glass bulbs, or polymeric bags.

Samples of soil gases in canisters, glass bulbs and polymeric bags are stable over long periods and multiple aliquots can be analysed from the same container.

b) Preconcentration onto solid sorbents.

Among the major advantages of using solid sorbents for trapping of hydrocarbons (SPE method) from soil gases belong:

- procedure is simple and cheaper than the other mentioned above,
- hydrocarbons trapped on sorbent are preconcentrated.

(2) GC-FID and/or GC-MSD analysis of hydrocarbons in trapped gasses or eluates obtained by desorption of hydrocarbons from sorbents using thermal or solvent desorption procedures. In very complex samples GC-FTIR, or GC-AED are used for identification purposes.

The quantitative analysis of petroleum hydrocarbons in environmental samples by CGC-FID in general is based on the assumption that the mass response of FID for hydrocarbons is constant and therefore any petroleum distillation fraction can be used as a reference mixture for calibration purposes. It shall be shown in this paper that the mass responses of FID for higher boiling hydrocarbons are not constant. It has been moreover found that mass response calibration factors for petroleum oils boiled in the equal temperature interval differs more than 2.5 fold in dependence on the hydrocarbon group composition. For the CGC-FID determination of such petroleum hydrocarbons in environmental samples it is therefore necessary to use a reference material which provides not only chromatograms but also the group composition similar to that in the considered samples.

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