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## PROBLEMS WITH THE DETERMINATION OF PETROLEUM HYDROCARBONS IN ENVIRONMENTAL SAMPLES

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Methods for determining the content of total petroleum hydrocarbons (TPH) in environmental samples are based on extraction of TPH from environmental samples and determination of THP in obtained extract by (i) gravimetric method, (ii) gas chromatography (GC-FID or GC-MSD) and (iii) infrared (IR) spectrometry.

The gravimetric methods measure the weight of total extracted contaminants by means of the analytical balance. Groups of substances with similar solubility in an organic extracting solvent are determined quantitatively. There is no need to calibrate this method. The concentration of specific petroleum constituents is not determined by this method.

The infrared (IR) spectrometric methods measure the adsorption of C-H bonds in the 3200 to 2700 wave number ranges. Quantitation is achieved by comparing IR absorption of the sample against that of known oil (e.g. a sample of the spilled product) or a reference oil. However, there are some inherent problems with the IR based methods. All petroleum hydrocarbons do not respond equally to infrared analysis, and comparison of the unknown to the standard mixture may result results with high systematic error.

The gas chromatographic (GC) methods are based on comparison of a standard chromatogram response (the overall peak area) to the chromatogram response of the samples.

In general GC-FID is preferred over IR for the determination of petroleum hydrocarbons in environmental samples. The main reasons for these preferences are:

- lower detection limit,
- capability to identify individual hydrocarbon components in addition to total petroleum hydrocarbons,
- less interference by naturally occurring substances. In this respect capillary GC-MS is the method of choice.

Weathering can affect both GC and IR analysis of petroleum hydrocarbons in environmental samples. A chromatogram of a fresh petroleum distillate fraction can be very different from that a weathered sample. There will be a depletion of some straight chain alkanes in the weathered sample compare to the fresh sample.

The aim of this paper is to show problems connected with the calibration of the determination of C<sub>20</sub>-C<sub>44</sub> petroleum hydrocarbons by GC-FID and IR spectrometry. It shall be shown that both GC-FID as well as IR spectrometric method requires to use of standards which group composition is matched with that present in the environmental samples.

### Reference

*Analysis of Soils Contaminated with Petroleum Constituents*, O'Shay T.A. and Hoddinott K.B., Eds.; ASTM PCN 04-012210-38, Philadelphia, PA, USA, 1994.