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Application Note #AZ3004

A Simplified Procedure for the Determination of TPH's in Waters and Soils Using the HC-404 System

The determination of Total Petroleum Hydrocarbons (TPH), greases, and lubricant residues are important environmental and industrial tests. Fuel and lube oils can be determined in water and soils to track spills and storage tank leaks, while many military and industrial situations require certain components (gas regulators, space shuttle components, weaponry parts, etc.) to have a maximum limit of grease or oil on them to prevent potentially hazardous conditions.

These tests are currently done by extracting and washing, respectively, the materials being tested with Freon-113 (Dupont), or equivalent non-hydrocarbon solvent. The extract/wash liquid is placed in a cell and measured in a filter infra-red photometer optimized for ~2940 cm^{-1} (or ~3.4 microns). Regular Freon Chloro-Fluoro-Carbons have no absorbing bands here, so any signal has to come from the hydrocarbons that are extracted into the Freon.

There are many specific procedures defined by such agencies as the **U.S. EPA** (Methods #418.1 / 413.2), **ASTM** (Methods for Petroleum Hydrocarbon contamination in Water, Soil and Machinery), **AOAC** (Oil contamination in plants & processed food materials), and **IUPAC** (International methods). Most states and some municipalities have modifications on these standard methods. Since the methodology defines only a relative measure of total hydrocarbons, it is understood that a sample extraction procedure needs to be more reproducible than accurate. Using a compilation of various techniques mentioned above, we have detailed two (2) simplified procedures for the analysis of TPH in water samples (waste, potable, natural) and soils (sandy, loam-rich, high-clay). These can be used as a reproducible starting point for properly and accurately characterizing the sample material, based on your regional requirements.

Examples of several sample extractions, with statistical calculations on precision and detectability, are shown on the reverse. The analyses were performed using the Buck HC-404 Fixed Filter Infra-red Analyzer. Comparisons are made to results from the classic gravimetric technique using Freon and a rotary flask evaporator, and to quantitative data obtained from an FTIR spectrophotometer. A calibration curve was made using a Freon Blank, 10 and 50ppm standards, using the Buck 404-11 EPA-Style Reference Oil.

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Samples: (1) Waste-Water from industrial machine shop
(2) Soil from 8' deep drilling on residential property
(adjacent to abandoned Gas Station, ~15 years old)

Preparation: (1) Collect 1 liter water in a 32oz Nalgene HDPE bottle, add 10ml concentrated HCl and 20ml Freon-113. Shake well for 1 minute, let settle and separate, pipet Freon from bottom. Gives a 50:1 concentration.
(2) Place 30 grams of soil in 4oz Nalgene wide-mouth HDPE bottle, add 30ml 20% HCl in water and shake to mix. Add 30ml Freon-113, re-cap and shake well for 2 minutes (Paint shaker can be used for multiple samples). Let settle and pipet Freon from top of soil. If separation of the layers is not clear, add some Sodium Sulfate to break the emulsion. Gives a direct (1:1) extraction.

Calibration: A Freon-113 BLANK (0ppm), a 10ppm and 50ppm standards were used to create a linear curve on the HC-404 unit.

Analysis: (1) Buck Model HC-404 with standard 10mm IR Quartz Cuvette
(2) Haake Roto-Vap with 125ml round-bottom flask
(3) Nicolet medium resolution FT-IR

Results: Values are mg'L (ppm) Oil in the original samples:

	Water / RSD / D.L.	**Soil / RSD / D.L.**
Filter IR	7.9 / 3% / 0.2	27.5 / 2% 7.0
Gravimetric	7.4 / 5% / 1.0	24.2 / 7% / 10.0
Scanning IR	7.8 / 4% / 0.5	27.8 / 5% / 25.0

Correlation Coefficient of Calibration Standards = **0.993**

Environmental labs and industrial facilities can answer many of their contamination questions by using the Model HC-404 as the primary tool for oil and petroleum hydrocarbon testing. The Buck HC-404 provides a very rapid and adaptable technique for many types of samples. The open-design sample compartment allows a variety of cells to be used for optimizing signal response, and the unique narrow-band optical filter provides superior stability and signal-to-noise ratios that match complicated, more costly FTIR systems. Overall performance of this unit is ideal for all types of bulk hydrocarbon analyses.

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