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ANALYTICAL METHODS FOR QUANTITATIVE AND QUALITATIVE DETERMINATION OF HYDROCARBONS AND OIL AND GREASE IN WATER AND WASTEWATER

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ABSTRACT

Analytical methods to determine oil and grease concentration and identify specific organic fractions in water and wastewater are reviewed. Important aspects of the development of these procedures are extraction technique, solvent type, and identification and quantification methods for the extracted material. The material presented will assist researchers and regulatory investigators in selecting appropriate analytical procedures and interpreting results.

INTRODUCTION

Oil and grease analysis, like many analytical methods for determination of water quality, does not measure a specific substance or compound. Oil and grease analyses attempt to quantify compounds which have a greater solubility in an organic solvent than in water. The principal types of compounds included in oil and grease analyses are fats, soaps, fatty acids, hydrocarbons, waxes, and oils. The contribution of each of these substances will depend upon the origin of the wastewater being analyzed and the type of extracting solvent used.

Discharge regulations in the United States typically include measurements of oil and grease. While imposing relatively simple analytical requirements, oil and grease tests result in measurement of a broad range of compounds with widely varying chemistry and toxicity. Methods are available for identification of specific organic fractions but tend to be too demanding of expertise, time and equipment to be used as a regulatory tool. The review presented in this paper was prepared as part of a larger study evaluating techniques that are suitable for routine analysis while providing useful information about the source and type of organic compound.

EARLY ANALYTICAL METHODS

The development of analytical techniques to measure oil and grease was initiated by wastewater treatment plant personnel who found that large quantities of grease clogged or detracted from the performance of treatment plants. Numerous examples of anaerobic digester failure were attributed to the build-up of greasy scum layers which prevented gas transfer and mixing. Other examples include failure of sewers due to the build-up of grease deposits. These failures were frequent at rendering plants and slaughter houses. Treatment plant operators were faced with a need to measure oil and grease concentration in order to prevent process failure by preventive maintenance or control measures.

The earliest documented analytical method for oil and grease analysis was made by Hazen (1). Hazen's method was used until the early thirties when other methods were proposed. Basically, Hazen's method requires that a 500 ml. sample of oily wastewater be evaporated to 50 ml, neutralized with hydrochloric acid, evaporated to dryness, and extracted with a solvent. The solvent is then evaporated to dryness in a tared container which is weighed to determine residue. The residue is reported as oil and grease.

Knechtges, et al. (2) reported a modified procedure which uses a Caldwell extractor to extract the residue resulting from evaporating the wastewater sample. They investigated ethyl ether, petroleum ether and chloroform as solvents and found that chloroform gives the highest residue weight. They also investigated the types of compounds which are present in the extract, finding that free acids comprise as much as 76% of the oil and grease in primary effluents.

Hazen's method and the modification proposed by Knechtges are time consuming, requiring as much as two days to perform the analysis and often produce inconsistent results. The methods were unacceptable and new methods were developed by numerous investigators, summarized as follows:

1. Alum coagulation, filtration, drying of the filtered material and sixteen hour extraction with petroleum ether (3).
2. Acidification and refrigeration, filtration, extraction of the filtered material, and drying (4,5).
3. Liquid/liquid extraction of an acidified sample, separation and drying of the extract (6).
4. Lime coagulation, filtration, modification of filtered material, fluffing, and extraction for four hours (7).
5. Oil extraction of sewage solids or sludge and extraction of the oil (8).

All these methods use petroleum ether as the solvent and require weighing of the extract to obtain the oil and grease residue. The methods were designed to overcome the problems associated with Hazen's existing standard method. The most important of these problems is the lengthy time required to evaporate 450 ml of water. This step introduces error as well as inconvenience, since the low molecular weight fractions of the oil and grease are lost during evaporation. The evaporation step was not necessary in the newer methods.

Another problem with Hazen's standard method was its inability to extract a high percentage of the fatty acids, due to the formation of insoluble precipitates in drying at neutral pH. This problem is overcome by acidification of the filtered material, which converts the precipitates to organic acids. The third problem associated with Hazen's method, and a problem which still exists today, is extraction efficiency.

Hatfield and Symons (1) reviewed the six proposed methods with the objective of recommending a new standard method. They define grease as "that material which is extracted from an acidified sample of sewage by petroleum ether (b.p. 40° - 60C) when using the standard procedure as recommended by the committee." Obviously, this is a working definition which has a very tenuous relation to the scientific description of the compounds which comprise grease. They also indicate that all the methods are very useful and under optimum conditions all give reasonable results. For a standard method they propose the acidification technique (4,5) using petroleum ether as a solvent. The liquid-liquid extraction (6) gives low results when compared to the other methods, and was not selected.

MODERN ANALYTICAL METHODS

The adoption of the Okun-Ludwig method for oil and grease did not result in a satisfactory method, and development of improved methods continued. Pomeroy (9) proposed an improved liquid-liquid extraction method. Gilcreas et al. (10) reviewed all the methods (1) and proposed a modified method, which they called the modified Sanderson method. The Sanderson method was developed by Gilcreas and Sanderson, but was never published, and was a modification of the post-1945 standard method. The Sanderson method uses a slightly different type of filtration material, but is otherwise very similar to the post-1945 standard method. The modified Sanderson method, using Freon 113 (1,1,2 tri-chloro -1,2,2 trifluoroethane) as a solvent instead of petroleum ether, has survived and is one of the four current standard methods. Gilcreas et al. (10) found all alternative methods to the modified Sanderson method unacceptable because of the lengthy analytical procedures (approximately 25 hours).

The next development in oil and grease analysis modified the solvent used for extraction. Petroleum ether had been used almost exclusively it was shown (11) that n-hexane can be used as effectively as petroleum ether. This is not surprising since n-hexane comprises a large portion of petroleum ether. Normal hexane is a preferred solvent since it is more uniform in characteristics than petroleum ether. Unfortunately, n-hexane, like petroleum ether, is quite flammable, and for this reason other non-flammable solvents are needed.

Chanin et al. (12) proposed using Freon 113 as a solvent. They showed that its use gives results that are virtually the same as n-hexane, and overcomes the flammability problems. Taras and Blum (13) confirmed these results. More importantly Taras and Blum showed that extraction efficiency can be greatly improved if sodium chloride is added to the oil and grease sample at a concentration of 5 gL^{-1}. The use of salt overcomes the problem of low extraction efficiency with the liquid/liquid extraction. The high salt concentration apparently coagulates the emulsified oil and grease by double-layer compression.

The next important development was reported by Gruenfeld (14) who showed that the extracted oil and grease in the solvent can be measured by infrared spectrophotometry. He showed that this was possible because of light absorption at 2930 cm^{-1}. Absorption at 2930 cm^{-1} results because of the CH_2 bond which is a common characteristic of oil and grease.

The use of spectrophotometry overcomes two important problems with the oil and grease analysis. First, it extends the nominal limits of detection of oil and grease to levels below 10 mg/l^{-1}. Second, the evaporation of solvent is not required in the IR spectrophotometry technique, which reduces the loss of low molecular weight compounds, an unfortunate shortcoming of all the gravimetric techniques.

There are five methods for the quantitative determination of oil and grease in waters and sludges in the 1985 edition of *Standard Methods for the Examination of Waters and Wastewater.* The methods are summarized as follows:

1. 503A Partition Gravimetric Method
2. 503B Partition-Infrared Method
3. 503C Soxhlet Extraction Method
4. 503D Extraction Method for Sludge Samples
5. 503E Hydrocarbons

Methods 503A and 503B are modifications of liquid-liquid extraction (6) using Freon 113. The Freon 113 is evaporated after extraction and the residue is weighed. This method has the disadvantage of losing low molecular weight compounds in the drying step. Method 503B is identical to 503A except that the oil and grease in the extract is analyzed spectrophotometrically. Method 503B has the disadvantage of requiring an expensive instrument and the development of a calibration curve, often without knowing the types of compounds present in the extract. These compounds will generally not be known. As an approximation, *Standard Methods* recommends a reference oil standard, composed of 37.5% isooctane, 37.5% hexadecane, and 25% benzene. Method 503B is much more sensitive than method 503A and suffers less from loss of low weight hydrocarbons during drying.

Methods 503C and 503D are virtually identical to the Okun-Ludwig method adopted in 1945. They have the advantage that difficult to extract materials are often more easily extracted in the Soxhlet device.

Method 503E is not an independent method but is an extension of the preceding four methods, and allows separate analysis of hydrocarbons. Method 503E takes advantage of the polar nature of fatty acids, which enables silica gel to preferentially adsorb them. By contacting the oil containing extract from methods 503A, B, C, D with silica gel, the polar compounds can be removed, leaving only the non-polar fraction, which is generally composed of hydrocarbons. This method, in combination with method 503B, permits the rapid quantitative analysis of oil and grease as well as hydrocarbons. Techniques for separating and quantifying oil and grease fractions are discussed later.

Standard Methods recommends that Method 503B be used when low concentrations (less than 10 mg l^{-1}) are to be measured. For the analysis of sludges, very high concentrations, or very heavy petroleum hydrocarbons, Method 503C is recommended. Method 503E is recommended in combination with any of the other methods when it is desirable to determine the non-polar fractions.

The precision and accuracy of each method are also reported in *Standard Methods*. In general, the recovery of the methods is approximately 90 to 99% depending upon the type of oil present in the samples. Very low molecular weight compounds can be lost due to volatilization, while very high molecular weight compounds are often not recovered well in liquid/liquid extractions. The standard deviation of the methods is reported to be in the range of 0.9 to 1.4 mg/l when analyzing samples containing 12 to 17 mg/l^{-1} of oil and grease. *Standard Methods* indicates that Method 503C is the most subject to variability from technique.

A new analysis in the 15th edition of *Standard Methods* is a procedure for Freon-extractable floatables, Method 206B. It is a new method and is therefore a tentative method. The procedure is used to measure only the oil and grease portion which is free and floating at the surface of water. It uses a special oil flotation tube which resembles an extraction flask. Using this extraction flask it is possible to allow oil and grease to float to the surface for a specific period. After the flotation period the subsurface portion of the sample is discharged and the remaining material is measured as in Method 503A. The test is used to predict gravity oil-water separator performance and also as a measure of aesthetic qualities of water.

The choice of solvent remains an open issue. There are numerous reports in the literature of various researchers using solvents other than Freon 113. Other solvents which are used include chloroform, carbon tetrachloride, and n-heptane, n-pentane as well as n-hexane. Gruenfeld (14) compared Freon 113 with carbon tetrachloride and found that carbon tetrachloride is slightly more efficient for extracting No. 6 and No. 2 fuel oils and crude oil, but concludes that the increased efficiency of carbon tetrachloride does not overcome its increased health risks. Chloroform appears to be a better solvent than Freon 113 for extracting heavy oil or highly polar compounds (15). Meyers, et al. (16) compared both n-hexane and Freon 113 and found that they extract similar quantities of oil and grease, but that the variability in results is lower with Freon 113.

ASTM (17) also has identified techniques for oil and grease analysis, specifying three procedures using two different solvents (n-hexane and chloroform).

INSTRUMENTAL METHODS FOR AUTOMATING OIL AND GREASE ANALYSIS

Over the past five years there has been an increased interest in instrumental methods for quantitative determinations of oil and grease. This results primarily from the need for remote sensing, which can be used for remote detection of oil spills and process failure. The methods evaluated include fluorometry (18), light scattering (19,20) dye transfer (21), and light transmissions (22). Evaluations of several methods have been presented (23,24). An overview of these methods indicates that they are successful in many applications, but no method is applicable in all types of water.

TECHNIQUES FOR FRACTIONATING AND IDENTIFYING EXTRACTABLE ORGANICS

As indicated previously, oil and grease is composed of a variety of compounds. Consequently, analytical procedures have been developed to distinguish between hydrocarbon and non-hydrocarbon fractions which comprise oil and grease in an attempt to distinguish between biogenic and anthropogenic sources. Method 503E is an example.

A major environmental consideration in selecting a technique is the relationship between identified material and its toxicity. However, toxicity data on specific compounds is sparse and sometimes conflicting, and data on groups of compounds distinguished by extraction techniques or adsorption properties are almost entirely lacking. This is particularly true for data on chronic, rather than acute toxicity. Referenced here are some general observations which help distinguish toxicity among hydrocarbon fractions, and are useful as a guide to developing methodology. Aromatic hydrocarbons are generally more toxic than aliphatics, with the toxicity of aromatics increasing with the number of rings and with the degree of alkyl substitution. However, solubility decreases with increasing numbers of rings and alkyl groups. Thus, the most toxic petroleum hydrocarbons may be composed of 4-5 rings aromatics, although the most toxic contribution may be exerted by mono or dinuclear aromatics (25-28). As a result, techniques have been developed which attempt to distinguish between the aromatic and aliphatic fractions of oil and grease.

Most techniques to differentiate hydrocarbons from other oil and grease compounds use a solvent with selected properties, followed by some type of chromatographic technique to separate fractions by polarity or other property, such as silica gel chromatography. The separated fractions can be qualitatively analyzed by instrumental techniques such as gas chromatography. Additionally, a physical process such as filtration or centrifugation of samples prior to extraction is often practiced. An example of an analytical procedure to differentiate organic fractions which uses many of the procedures discussed in this review is shown in Figure 1 (29). The following sections describe solvent selection, fractionation technique and identification method.

Solvent Selection

The solvent selection is critical to the type of material extracted, as well as the extraction efficiency. The solvent should have a high solubility for the desired organics, a low miscibility with water and a low boiling point to facilitate removal of the solvent from the extracted material. Simple organic solvents, such as pentane and hexane, have been used to investigate the aliphatic hydrocarbon fraction in water (30-32). These solvents do not efficiently recover polynuclear aromatic hydrocarbons, triglycerides or polar compounds. When an organic fraction other than, or in addition to, aliphatics is to be extracted, other solvents have been used, such as dichloromethane (33-37), chloroform (15,38,39), petroleum ether (40), trichlorotrifluoromethane (41), benzene (29,42), and chloroform/methanol (1/1) (42). Choice of solvent depends largely on the desired fraction to be extracted and can significantly affect results.

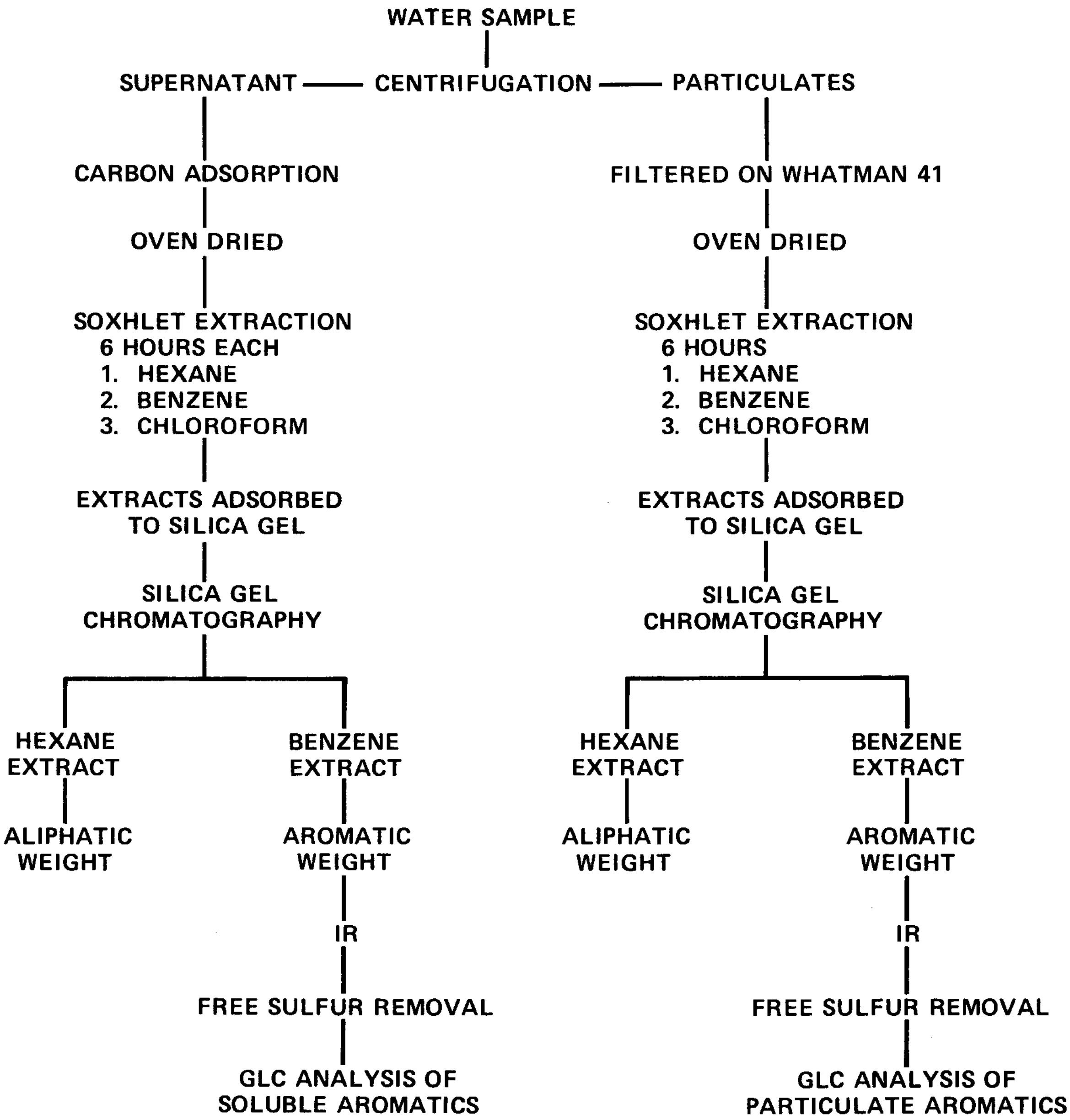

Figure 1. One Example of a Technique to Differentiate Extracted Material (1).

Separation Technique

Several chromatographic techniques are commonly employed to isolate and characterize hydrocarbon fractions. Column chromatography can be used to separate hydrocarbons in approximate order of polarity and molecular weight, which may be sequentially removed through the application of successive solvents (43). Column chromatography can be very valuable as a means of preliminary sample clean up. Thin Layer Chromatography (TLC) provides a convenient and inexpensive means of separating hydrocarbons from polar compounds (15). If the TLC plates are developed using multiple solvent systems in a two-dimensional fashion, significant separation between hydrocarbons and polar compounds can be achieved. In the best case hydrocarbons can be separated into distinct fractions, depending on degree of saturation and chain length.

Identification and Quantification Techniques

As indicated previously, gravimetric and infrared spectrophotometry (IR) provide the simplest methods for quantifying hydrocarbons. IR techniques generally result in higher results than gravimetric techniques, probably due to the loss of volatile materials during extract drying (44-45). Analyzing the infrared spectrum can provide information about the chemical makeup of the extract, which is an additional advantage of the IR technique. However, accurate calibration of the infrared spectrophotometer is impossible for a sample with unknown chemical makeup.

Alternatively, the extract can be quantified or examined by UV-Visible spectrophotometry. Since electron state transitions for alkanes occur at a wavelength below 200 nm., which is difficult to analyze, UV-Visible analysis is most suited for conjugated systems, which will absorb at wavelengths greater than 200 nm.

Fluorescence spectrophotometry has been used to quantify aromatic hydrocarbons both through the examination of extracts and through direct water sample examination. Fluorescence spectrophotometry has the potential to rapidly differentiate 2 and 3 ringed aromatic structures from compounds with a greater number of aromatic rings.

Gas chromatography has regularly been used by researchers to separate, identify and quantify hydrocarbons using Flame Ionization detectors. Gas chromatography and/or GC/MS have become the methods of choice in instances where component identification of a hydrocarbon mixture is desired. With the advent of capillary columns it has become commonplace to separate and identify hydrocarbon isomers.

Mass spectrophotometry, used in conjunction with gas chromatography, allows for more definitive identification of the compounds separated by gas chromatography. Comparison of the sample MS spectrum with the spectrum of the pure postulated compound under identical chromatographic conditions increases the certainty of identification. Unfortunately, the combination of a GC/MS is not usually as sensitive as a GC using a hydrogen flame ionization detector.

The recent advances in high resolution pulse Fourier transform ^{13}C nuclear magnetic resonance spectrophotometry (NMR) was a major development in the analysis of organic fuels, due to increased sensitivity and resolution. ^{13}C NMR has increasingly been used in the analysis of complex systems such as oils, proteins, and nucleic acid chains. Although the identification of specific compounds in an oil and grease sample may be more difficult by NMR, structural characteristics about the overall extract may be deduced. General structural characteristics, such as aromatic versus aliphatic differences, and extent of hydroxylation or presence of halogen atoms is a useful indicator of environmental significance.

High pressure liquid chromatography (HPLC) is able to provide useful separation of organic compounds, and is particularly valuable for compounds which have high boiling points, or are unstable at high temperatures. A variety of detectors are available for HPLC, and analytical procedures can be developed and optimized for particular applications. HPLC, like the other instrumental techniques, require considerable expertise and expenditure of resources.

ALTERNATIVE METHODS FOR DETERMINATION OF GREASE-LIKE COMPOUNDS

There are other procedures which measure the total organic content of a sample which might be useful for oil and grease analysis. Total organic carbon, total oxygen demand, and chemical oxygen demand analyses may be useful indicators of oil and grease content. Their utility will depend almost entirely upon the origin of the water. Wastewaters from processing operations where oil and grease is the single or predominant contaminant would be good candidates for trial use. Wastewaters from domestic origin would contain high concentrations of non-oil and grease organics such as carbohydrates and proteins, and would be poor candidates. Urban stormwater might be a good choice for TOC analysis, depending upon the land-use creating the stormwater.

SUMMARY AND CONCLUSIONS

The development of oil and grease quantitative analysis has been reviewed from the earliest known method to the current standard methods. Also reviewed are methods to separate organic fractions and identify compounds contained within the definition of oil and grease. A summary of available techniques is shown on Table 1, which includes a brief description of the advantages and disadvantages of each technique.

Oil and grease analyses measure a group of compounds which have a common characteristic: solubility in a particular organic solvent. Gravimetric and infrared (IR) spectrophotometric techniques provide the simplest quantification methods. Infrared techniques generally result in higher readings than gravimetric techniques, possibly due to the loss of volatile materials during drying (44-45). Analysis of the infrared spectra can give information about the chemical make-up of the sample, which is an additional advantage of the IR technique. However, accurate calibration of the infrared spectrophotometer is impossible for a sample with unknown hydrocarbon constituents. Infrared analysis may be preferred for low concentrations, or when a quick or automated procedure is required. Standard gravimetric analysis of the extract is possible for high concentrations (greater than 10 mg L^{-1}), and where volatilization of the low molecular weight hydrocarbons is not important.

Qualitative methods to identify specific organics include GC, HPLC, GC/MS methods and IR/UV/Visible spectrophotometric techniques. Fractionation of the extract by column chromatography or TLC assists in compound identification, and can be used to quantitate selected fractions, such as aliphatic hydrocarbons. However, these techniques are far from routine at present, and their applications will probably be limited for regulatory purposes, due to cost.

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Table 1. Summary of Hydrocarbon Analysis Techniques

Technique	Equipment Requirements and Availability	Level of Laboratory Personnel	Time for Analysis (Following Preliminary Extraction)	Cost of Representative Equipment	Analytical Capabilities and Limitations
Gravimetric	Balance. Available in almost all laboratories.	Little training required. Technician level.	Minutes/sample.	Balance $3,000-$8000	Quantification of extracted materials. Some loss of volatile materials.
Infrared spectrophotometry	Infrared spectrophotometer. Available in many laboratories.	Some training required for operation and standard preparation. Entry or journeyman level chemist.	Minutes/sample following instrument set-up calibration and standard preparation	$5,000-$25,000	Difficult to establish reference oil for calibration.
UV and visible spectrophotometry	UV/visible spectrophotometer. Available in many laboratories.	Some training required for operation and standard preparation. Entry or journeyman level chemist.	Minutes/sample following instrument set-up calibration and standard preparation.	$5,000-$25,000	Detection and quantification of extracted material with potential for some qualitative assessment by trained personnel.
Column chromatography	Chromatography columns and packings. Not common, but easily obtained.	Not routine procedures. Entry or journeyman level chemist.	Several hours/sample, although not requiring constant attention.	Chromatography tubes $17-34 (10 mm ID) Silica gel column sorbents $32-72 (500 grams).	Fractionate sample. Detection and quantification subsequently done by other techniques. Useful for preliminary sample cleanup.
Gas chromatography	Gas chromatograph with suitable detector. Available in many laboratories. Mass spectrometer detector not commonly available, but easily obtained.	Considerable training required for comprehensive analysis. Journeyman or senior level chemist.	Highly variable depending on specific technique. At best, limited to a few samples/day.	$5,000-$20,000 for a GC, other significant set-up costs.	Fractionate sample with potential for sensitive identification and quantification. Usually needs preliminary sample cleanup.

Table 1. Summary of Hydrocarbon Analysis Techniques (Continued)

Technique	Equipment Requirements and Availability	Level of Laboratory Personnel	Time for Analysis (Following Preliminary Extraction)	Cost of Representative Equipment	Analytical Capabilities and Limitations
Nuclear Magnetic Resonance Spectrophotometry	Nuclear magnetic spectrophotometer. Not common laboratory equipment. Obtainable from a few manufacturers.	Considerable training required. Journeyman or senior level chemist.	Variable, but limited to a few sample/day. Complex preliminary analysis required.	>$20,000	Useful in characterizing chemical structure.
Thin layer chromatography	Thin layer chromatography plates, developers, and method for detection. Available in many laboratories and easily obtained.	Some training required. Entry or journeyman level chemist.	Variable, but relatively rapid technique.	Glass pre-coated silica-gel plates $44-150 (package of 25).	Useful in separating fractions. Not comparable to other techniques.
High pressure liquid chromatography	High pressure liquid chromatograph. Not common laboratory equipment. Obtainable from a few manufacturers.	Considerable training required. Journeyman or senior level chemist.	Highly variable depending on specific technique. At best, limited to a few samples/day.	>$20,000 significant set-up costs.	Complements GC techniques for identifying compounds. Quantification subsequently done by other means.
Fluorescent spectrophotometry	Fluorescence spectrophotometer. Not common laboratory equipment. Available from a few manufacturers.	Not routine procedure. Journeyman level chemist.	Rapid following equipment set-up and calibration. Minutes/sample.	$3,000 and up.	Potential for use for qualitative analysis not adequately explored. Useful as a means of detection.

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