

CHEMICAL CHARACTERIZATION OF FRESH, USED AND WEATHERED MOTOR OIL VIA GC/MS, NMR AND FTIR TECHNIQUES

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ABSTRACT. Motor oil undergoes a range of chemical and physical transformations during routine engine operation, and oil transformations continue following release to the environment. Some components of motor oil, particularly used oils, are considered a threat to public health and the environment. Gas chromatography-mass spectroscopy (GC/MS), Fourier-transformed infrared spectroscopy (FTIR), nuclear magnetic resonance spectroscopy (NMR), and flame atomic absorption spectrophotometry (FAAS) techniques were employed to characterize the chemical composition of fresh, used, and weathered used oil samples. Used oil was weathered by adding to soil at a rate of 1.5% (w/w), seeding with mixed grass and legume species, and incubating. Soxhlet-extractable oil was analyzed over 150 days. Compared with fresh motor oil, used oil contained new aliphatic and aromatic hydrocarbon compounds such as 1,3,5-trimethyl benzene, *p*-xylene and methyl ester undecanoic acid. FTIR bands at 1704–1603 cm^{-1} were related to the presence of carbonyl groups, and bands at 869, 813 and 1603 cm^{-1} were associated with new aromatic hydrocarbons including polycyclic aromatic hydrocarbons. NMR analysis revealed new peaks in the used oil in the range 2.1–2.7 ppm and 6.8–7.2 ppm, which were associated with new aliphatic and aromatic hydrocarbon products, respectively. FTIR and GC-MS analysis of weathered used oil indicated the presence of various alcohols, aldehydes and ketones, indicating substantial biological and chemical decomposition. It is not known, however, if detoxification of the used oil occurred.

Keywords: Fourier-transformed infrared spectroscopy, gas chromatography, hydrocarbons, nuclear magnetic resonance spectroscopy, used motor oil

From the mid-twentieth century onward, the standard of living in the United States and many other industrialized nations increased markedly. This period also experienced a corresponding increase in motor vehicle use. A significant drawback of increased motor vehicle use has been contamination of soil and groundwater with crankcase oil, with consequent impacts on ecosystems and public health.

Efforts to decrease the volume of used motor oil generated have engendered encouraging results. Approximately 800 million gallons (3,000,000,000 liters) of used oil are recycled annually for reuse (U.S. EPA 2001). In the 1970s, as a result of the energy crises, recycling used oil programs and do-it-yourself oil-changing and recycling were encouraged (U.S. EPA 1989). However, significant amounts of used motor oil continue to be discharged to the environment. The U.S. generates 63% of the estimated 1.4 billion gallons (5,300,000,000 liters) of used motor oil generated annually (Blodgett 1997), and it is es-

timated that 30 percent may not be disposed of properly. Ninety percent of oil filters from do-it-yourself oil-changing, which contains 10 million gallons (38,000,000 liters) of used oil, are not recycled but dumped into trash, with the potential of polluting soil and water (IDEM 2002).

Used motor oil may contain minute quantities of gasoline, additives (detergents, dispersants, oxidation inhibitors, rust inhibitors, viscosity improvers), nitrogen and sulfur compounds, a broad range of aromatic and aliphatic hydrocarbons with chain lengths ranging from C_{15} to C_{50} , and metals such as lead (Pb), zinc (Zn), calcium (Ca), barium (Ba) and magnesium (Mg). These contaminants arise from normal wear of engine components and from heating and oxidation of lubricating oil during engine operation. Used oil may contain higher percentages of polycyclic aromatic hydrocarbons (PAHs) and additives compared to fresh oil (Hewstone 1994; Vazquez-Duhalt 1989). The concentration of PAHs in used oil may range from 34 to 190 times higher than

those in fresh motor oil (Grimmer et al. 1982). Naphthalene, acenaphthalenes, dinaphthenoanthracenes benzo[*a*]pyrene, and benzo[*a*]anthracene were detected in used oil samples by Cotton et al. (1977) and ATSDR (1997). PAH compounds represent a direct hazard to the environment and human health (Vazquez-Duhalt 1989; Hewstone 1994).

Analytical characterization of oily substances is hindered by the complex chemical structure of oil. Characterization by solubility provides information regarding its chemical nature, but not chemical structure (Moschopedis & Hawkins 1981). Techniques such as open column liquid chromatography (LC), medium pressure LC, and supercritical fluid extraction separate oil compounds by classes followed by characterization via other techniques (Barman et al. 2000). Techniques using mass spectroscopy (MS) have been more successful in oil characterization (Dilts 1998). Grimmer et al. (1982) used glass capillary GC-MS for PAH inventories of lubricating oils.

Many petroleum-based hydrocarbon substances are further degradable after disposal (Cole 1994; Cunningham et al. 1996; Heinonsalo et al. 2000; Banks et al. 1999; Shimp et al. 1993). Unfortunately, there is little information regarding the chemical transformations that such compounds, particularly motor oils, experience under natural weathering conditions.

Limited studies in the literature have assessed the chemical composition of automotive lubricating oils; however, many of these papers are dated and new oil manufacturing technologies, including the use of new additives, combined with different engine types and operations will result in the production of new wastes.

The objective of the reported study is to elucidate the chemical composition of fresh motor oil and compare it to used motor oil and used oil exposed to natural weathering processes. Analytical techniques including gas chromatography/mass spectroscopy (GC/MS), Fourier-transformed infrared spectroscopy (FTIR), nuclear magnetic resonance spectroscopy (NMR) and flame atomic absorption spectrophotometry (FAAS) techniques were applied for the analysis of oil samples.

METHODS

Used motor oil was collected and combined from several automobile crankcases. For comparison, four fresh motor oil samples, Castrol[®], Havoline[™], Pennzoil[™] and Mobil[™] (all 10W-40 weight) were obtained.

Gas chromatography/mass spectroscopy (GC/MS).—One drop of motor oil was placed in a screw-top vial (2 ml capacity) and diluted with hexane (HPLC grade) to 1 ml. The run consisted in the injection of 1 μ l of sample by a CTC A200S autosampler into a Varian model 3400 GC/INCOS50 MS. The GC was equipped with a polysiloxane capillary column 30 m in length, 0.25 mm internal diameter and 1.00 μ m film thickness. Helium was the carrier gas. The temperature program consisted of a heating rate of 12° C/min from 45° C to 325° C with a hold time of 10 min. An ASTM D2887 calibration standard was prepared at a 20 μ g/ml concentration. Data were collected on a Windows-based personal computer. Chromatographs were analyzed by the EnviroLink[™] program and a library search was used for identification of chromatographic peaks. Prior to the injection of samples the GC capillary column was cleaned by injecting a 1 μ l sample of hexane (HPLC grade).

Fourier-transformed infrared spectroscopy.—Sodium chloride (NaCl) polished discs (25 \times 2 mm) were used to analyze oil samples under Fourier-transformed infrared spectrometry (FTIR). Background spectra were obtained by scanning two clean discs simultaneously in the instrument. One drop of used oil was placed on one NaCl disc and was covered with a second NaCl disc. Both discs were placed in a Perkin-Elmer Spectrum 1000 FTIR instrument. Four scans were carried out in the 4000–400 cm^{-1} range.

¹H Nuclear magnetic resonance spectroscopy.—¹H NMR experiments were carried out on both fresh and used motor oil. A drop of oil was placed in a NMR tube. Deuterated chloroform (CDCl_3) was added to 5 ml of the oil sample and placed in a JEOL Eclipse +400 NMR instrument. NMR data and spectra were processed by the Delta NMR processing and control software, Version 3.1. A presaturation experiment was applied to decrease an intense signal and observe the baseline in detail. Sixteen scans were carried out, and the relaxation time was reduced from 4 sec to 3 sec.

Atomic absorption spectrophotometry.—

To determine the total metal content of the used oil, 0.5 g oil was placed in a digestion flask and 4 ml of concentrated H₂SO₄ added. The mixture was placed in a Hach DigestahlTM apparatus; a fractionating column placed at the top of flask acted as a vacuum, removing the acid vapor produced by refluxing. After 4 min refluxing at 440° C, 10 ml of H₂O₂ were added via a capillary funnel and refluxed for an additional 2 min (Hach 1989; Baker & Suhr 1981). After the reflux solution was cooled, deionized H₂O was added to bring the solution to 100 ml. Concentrations of Ba, Cd, Cr, Pb and Zn were measured using a Perkin-Elmer 2280 flame atomic absorption spectrometer (FAAS).

Weathering study.—*Soil collection and greenhouse setup:* Soil was collected from agricultural fields in east-central Indiana. The soil, a Glynwood loam (fine, illitic mesic Aquic Hapludalf), was transported to the laboratory and air-dried, finely ground with a mortar and pestle, and sieved through a 2 mm mesh sieve.

Ceramic pots were prepared containing 1 kg soil and amended with 1.5% (w/w) used motor oil. The used oil and soil were thoroughly mixed using a stainless steel stirring rod. Pots were seeded with a mixture of plant species including mixed grasses (creeping red fescue, *Festuca rubra*; fawn tall fescue, *F. arundinacea*; perennial ryegrass, *Lolium perenne*); and mixed clover (red clover, *Trifolium pratense*; ladino clover, *T. repens*). Pots received a pre-plant incorporation of a commercial 10–10–10 fertilizer.

Pots were watered to maintain the soil moisture content at approximately field capacity. Plants were grown for 150 days under natural lighting in the greenhouse.

Analysis of used oil in the root zone: Soxhlet extraction was utilized to extract oil hydrocarbons from soil after 100 days incubation with plants. Sodium sulfate was purified by drying overnight in an oven at 150° C. Round Soxhlet flasks were dried at 105° C for 30 min. After cooling, the weight of the round flask and boiling chips was recorded. Ten grams of contaminated soil were mixed with 10 g dry Na₂SO₄ and placed in a cellulose extraction thimble (24 mm outer diameter × 65 mm length). Glass chips were placed at the surface to prevent the soil from escaping from

the thimble during extraction. A 300 ml volume of hexane was added to the flask and extracted for 5 h at 70° C (U.S. EPA 1996a).

The oil/grease obtained from Soxhlet extractions was recovered for analysis by dissolving in 9 ml hexane. Solutions were analyzed by GC/MS and FTIR. Operating parameters for both instruments are as shown above. All glassware was cleaned via acid washing, and, where appropriate, by CH₃Cl to remove oily residues.

RESULTS AND DISCUSSION

Gas chromatography.—The GC-MS spectra of hydrocarbon compounds occurring in both the fresh and used motor oil were expressed as broad bell-shaped curves from 24–40 min (Figs. 1, 2). The noise observed in this portion of the fresh oil spectrum hindered identification of many of the abundant peaks. Matching of GC peaks with the computer library ranged between 46–87%. The used motor oil GC/MS spectrum (Fig. 2) revealed numerous new and more intense peaks from 6–40 min compared to those in fresh oil. The most abundant peaks were located between 11–22 min. Likely compounds were identified as methyl ester undecanoic acid, 1,3,5-trimethyl benzene and xylene. Benzene-based compounds and naphthalene-related components are the predominant hydrocarbon structures in the composition of the used motor oil. Naphthalene, acenaphthalenes, dinaphthoanthracenes benzo[*a*]pyrene, and benzo[*a*]anthracene were detected in used oil samples by Cotton et al. (1977) and ATSDR (1997).

GC/MS spectra of oil recovered from the vegetated soil treatment at 100 days (Fig. 3) revealed aliphatic and aromatic peaks associated with nascent hydrocarbons, presumably formed by the biodegradation of used oil in the rhizosphere (root zone). After 100 days, methyl ester dodecanoic acid and methyl tetradecanoate were detected with a 94% match. Alcohols, aldehydes and organic acids are common products of the so-called 'beta-oxidation' sequence of long-chain aliphatic compounds. This is promising data, showing that potentially toxic oily wastes are being metabolized by native soil microbial populations. It is not known, however, if detoxification of the used oil occurred.

In addition, several unidentifiable com-

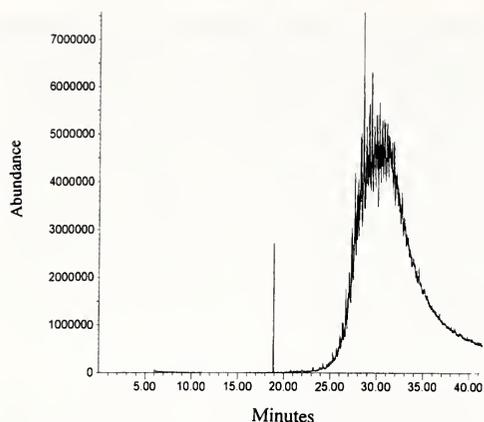


Figure 1.—Gas chromatography–mass spectroscopy (GC/MS) spectrum of a fresh sample of motor oil.

pounds occurred in the soil extracts. Based on previous studies, these may be branched chains (see Chaîneau et al. 1997). Additional studies are needed to further elucidate this so-called “unrecognized complex matter.”

Fourier-transformed infrared spectroscopy.—There were few notable differences in FTIR spectra between the four brands of fresh motor oil (Fig. 4); all samples possessed absorption bands in the same regions. In the used motor oil FTIR spectrum, new bands were observed at 1704.29 cm^{-1} and 1603.13 cm^{-1} (Fig. 5) indicating short-chain compounds with carbonyl groups from esters, ketones or acids (Kadam & Zingde 1985). These compounds are commonly formed in motor oil via chemical oxidation processes (Vazquez-Duhalt 1989). Organic acids, e.g., methyl ester undecanoic acid, were also identified by GC/MS. The used oil possessed a band at 1157.92 cm^{-1} (Fig. 5) which, according to Kadam & Zingde (1985), may correspond to peroxide compounds. Bands at 704.29 cm^{-1} and 1603.13 cm^{-1} are due to the presence of unsaturated additives (Zieba-Paulus & Koscielniak 1999). According to Rashid et al. (1990) and Kadam & Zingde (1985), bands at 1229 cm^{-1} and 724 cm^{-1} may be associated with the formation of nitrates from the oxidation of nitrogen oxide compounds.

Additives such as Zn, Ca and Mg salts or organic acids (i.e., sulphonate and phelonate) show bands in the range $600\text{--}1300\text{ cm}^{-1}$ and $1500\text{--}2000\text{ cm}^{-1}$ (Geach 1996; Kadam & Zingde 1985). A split band observed at 2925

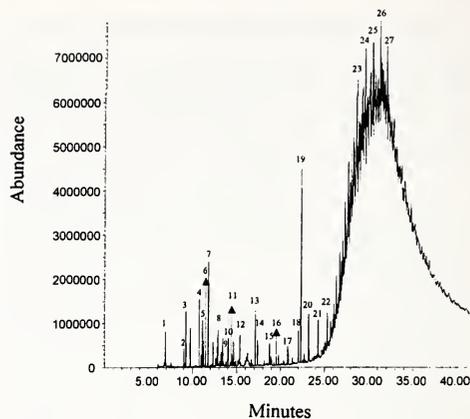


Figure 2.—Gas chromatography–mass spectroscopy (GC/MS) spectrum of a sample of used motor oil. Peak 1 = toluene; 2 = 1,2-dimethyl benzene; 3 = *p*-xylene; 4 = (2,2'-binaphthalene)-1,4,8'(5''H)-trione; 5 = 1,2,3-trimethyl benzene; 6 = 1-ethyl-2-methyl benzene; 1-ethyl-3-methyl benzene; 7 = 1,3,5-trimethyl benzene; 1,2,3-trimethyl benzene; 8 = 1-ethyl-3,5-dimethyl benzene; 1,2-diethyl benzene; 9 = 1,2,3,5-tetramethyl benzene; 10 = 1-methyl-3-(1 methyl ethyl) benzene; 1,2,3,4-tetramethyl benzene; 11 = 1-methyl-2-(2 propenyl)-benzene; 12 = naphthalene; azulene; 13 = 1-methyl-naphthalene; 14 = 2-methyl naphthalene; 1-methyl naphthalene; 15 = 1,1,3,3,5,5,7,7,9,9,11,11-dodecamethyl; hexasiloxane; 16 = decane; 5-methyl undecane; 17 = hexadecane; tricosane; heneicosane; 18 = eicosane; 2-methyl-tridecane; 19 = methyl ester undecanoic acid; methyl ester tridecanoic acid; 20 = 2-methyl tridecane; heptadecane; 21 = eicosane; phytane; 22 = hexacosane; dotriacontane; 23 = eicosane; 24 = 1-bromo pentadecane; 25 = eicosane; 26 = eicosane; 27 = eicosane.

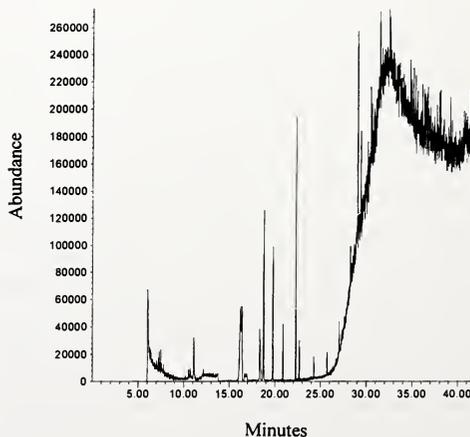
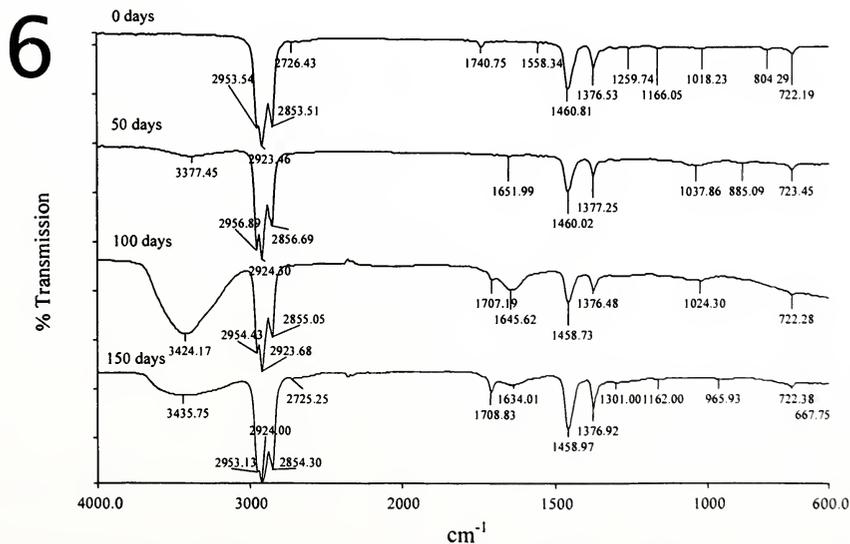
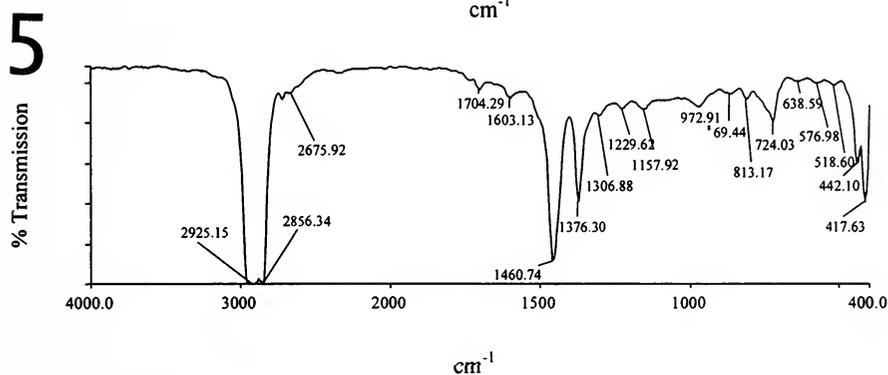
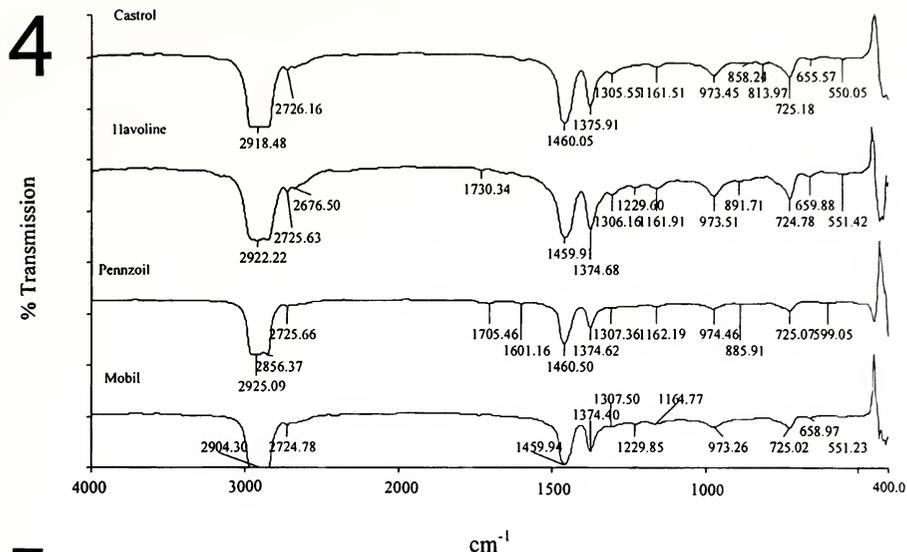
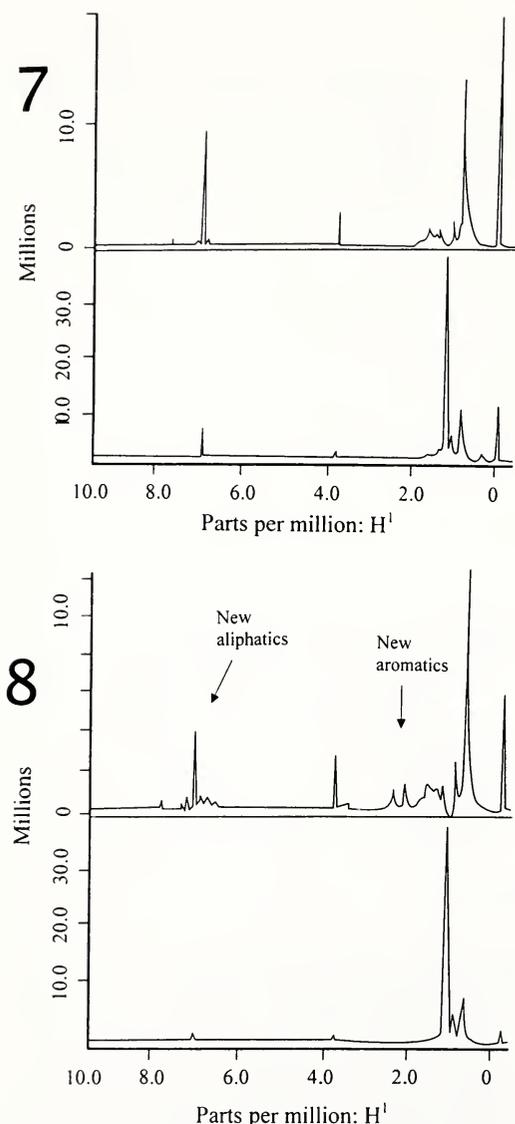


Figure 3.—Gas chromatography–mass spectroscopy (GC/MS) spectrum of a sample of motor oil from weathered soil after 100 days.



Figures 4–6.—Fourier-transformed infrared spectroscopy (FTIR) spectra of motor oils. 4. Four fresh motor oil samples; 5. Used oil; 6. Oil from weathered soil at 0 days (control), 50 days, 100 days, and 150 days.



Figures 7, 8.—Nuclear magnetic resonance spectroscopy (NMR) spectra of oils. 7. Fresh sample; 8. Used oil sample.

cm^{-1} , and bands at 1460 cm^{-1} and 1376 cm^{-1} occur due to the presence of a mixture of hydrocarbon compounds with small chain lengths and C–H branching vibrations within –CH– groups in the used oil.

There is evidence of the presence of aromatics and/or PAHs in the used oil since new bands appear at 869.4 cm^{-1} , 813.2 and 1603.1 cm^{-1} (Fig. 5), representing hydrocarbons in the aromatic ring (Geach 1996). On average, PAHs comprise about 4–8% of hydrocarbons

within used motor oils. Used crankcase oil commonly contains PAHs such as naphthalene, benzo[*a*]pyrene, and benzo[*a*]anthracene (ATSDR 1997). Cotton et al. (1977) reported that polyaromatic-polar compounds (i.e., acenaphthalenes, tetranaphthenophenanthrenes, dinaphthoanthracenes) accounted for 6.5% of a used motor oil sample.

FTIR analysis of oil residues over 150 days weathering in the plant root zone is shown in Fig. 6. Bands related to C–H vibrations appear in the range $2957\text{--}2850\text{ cm}^{-1}$. An intense band occurs at 1460 cm^{-1} and a less intense band at about 1377 cm^{-1} which may be produced by a mixture of compounds with small chain lengths and branching vibrations from C–H of the methylene (–CH₂–) chains in used oil. A broad and intense band occurred at 3424 cm^{-1} and a new band appeared at 1645 cm^{-1} , both of which are related to the O–H stretching band and the H–O–H bending vibrations of water. A band at 1740 cm^{-1} is associated with carbonyl groups in ketones, aldehydes and/or acids. Bands also appear at 1707 cm^{-1} and 1709 cm^{-1} and may be associated with new carbonyl groups arising from microbial oxidation of the oil in the rhizosphere. New bands occur at 1708 cm^{-1} and 1740 cm^{-1} which may correspond to new carbonyl-based compounds, possibly ketones or aldehydes generated by microbial oxidation processes.

Nuclear magnetic resonance spectroscopy.—Differences in NMR spectra were observed between the fresh and used motor oils (Figs. 7, 8). Used oil ¹H NMR spectra showed the presence of new aromatic peaks compared with new motor oil. New aromatic peaks were observed in the used motor oil between 7.2–6.8 ppm and new aliphatic compounds in 2.1–2.7 ppm range (Fig. 8). These new compounds may arise from the formation of PAHs, nitrogen-based compounds, i.e., nitrates (Kadam & Zingde 1985) and other substances, i.e., acids, esters and peroxides (Kadam & Zingde 1985; Geach 1996) formed during engine operation. NMR analysis was not carried out for the weathered hydrocarbons extracted from the soil.

Metal concentrations.—The used oil contained 838.0 mg/kg Zn, 110.0 mg/kg Pb, 9.4 mg/kg Cd and 4100 mg/kg Ba. Chromium was not detected. These numbers are substantially below that found in many earlier papers;

for example, Raymond et al. (1975) measured 7500 mg/kg Pb, 260 mg/kg Fe, 1500 mg/kg Zn, 17 mg/kg Cu and 21 mg/kg Cr in used oil. Cotton et al. (1977) reported, in 30 used oils, concentrations of 13,885, 655, 2500, 56, and 24 mg/kg of Pb, Fe, Zn, Cu and Cr, respectively. Vazquez-Duhalt (1989) measured 7000 mg/kg Pb, 1100 mg/kg Zn, 28 mg/kg Cu, 10 mg/kg Cr and 1 mg/kg Ni. The Agency for Toxic Substances and Disease Registry (ATSDR 1997) measured 240 mg/kg Pb, 480 mg/kg Zn, 3 mg/kg Cd, and 6.5 mg/kg Cr. The varied metal distributions reported for used motor oil samples are a function of total engine operating time and mechanical condition of the engine. Furthermore, the elevated Pb concentrations reported in earlier papers is typically the result of contamination by leaded fuels.

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