

ANALYSIS OF OIL AND GREASE COMPONENTS TO ASSESS THE QUALITY OF URBAN RUNOFF

M.K. STENSTROM¹, S. FAM¹, and G.S. SILVERMAN²

¹Water Resources Program, Civil Engineering Department, University of California
Los Angeles, California 90024 U.S.A.

²Association of Bay Area Governments, Oakland, California 94604 U.S.A.

Abstract – A technique modified from standard “oil and grease” testing procedures was used to examine runoff entering San Francisco Bay. This technique was developed to identify “oil and grease” fractions to provide a better understanding of environmental significance than indicated by “oil and grease” measurements. Samples were taken from fifteen different watersheds representing a variety of land uses. Collections were made during dry and wet weather conditions to represent the yearly diversity of water quality conditions. Organics were extracted with methylene chloride, virtually an identical approach to standard technique. Additional procedures were used to separate and quantify four fractions: non-aromatic, aromatic, polar, and very polar. The aromatic and aliphatic fractions were examined using high resolution capillary gas chromatography.

“Oil and grease” concentrations ranged from a low of 1.0 mg·l⁻¹ to as much as 20 mg·l⁻¹. Dry weather “oil and grease” discharge was considerably less than wet weather discharge. “Oil and grease” from land associated with commercial and industrial activity had as much as 60% hydrocarbon fraction, while “oil and grease” from residential and undeveloped areas were composed primarily of polar, non-hydrocarbon compounds. Correlations between land use and water quality parameters were examined. Aliphatics were associated with commercial land use. The percent of the aliphatic fraction composed of n-alkanes of C₂₅ and larger was inversely related to commercial land use. The aromatic fraction was more variable, with samples from non-commercial stations sometimes having very high aromatic fractions.

Keywords – “oil and grease” analysis, urban runoff quality, land use, water quality.

INTRODUCTION

The control of discharge of organic materials into the environment is hampered by the lack of convenient and inexpensive analytical techniques that reflect the potential impact of the pollutant. Although there is considerable research activity in the development of analytical methods for examination of oil in fresh water, as evidenced by numerous reports in the literature, very little work is directed toward the development of simple techniques. Most of the analytical procedures being proposed require considerable expertise and sophisticated equipment.

Regulatory requirements in the United States are largely based upon measurements of “oil and grease”, or freon extractable hydrocarbons. This procedure imposes relatively simple analytical requirements, but it also results in measurement of a broad range of compounds with widely varying chemistry and toxicity. The objective of this study was to demonstrate an analytical technique that is suitable for routine measurement, while providing more insight to potential environmental effects than provided by standard “oil and grease” measurements. We evaluated the technique’s utility through examination of urban runoff. A second objective was to relate land use to molecular composition of “oil and grease”.

This investigation was part of a larger study to determine the impact of non-point source pollution from urban areas on San Francisco Bay. A growing body of evidence indicates that organics present in runoff significantly impact the Bay. Previous work has shown that "oil and grease" concentrations in urban runoff from a typical mixed-use watershed in the Bay Area are frequently much higher than allowed for point source discharges, and indicates that runoff may be an important source of toxic organics (Stenstrom and co-workers, 1984).

BACKGROUND

DiSalvo and Guard (1975) and DiSalvo and co-workers (1975, 1976) have reported that aromatic hydrocarbons are present in fish and shellfish tissue, open water, algae and sediments in San Francisco Bay. Monoaromatic hydrocarbons have also been suggested as a major contributing factor to the current decline of the striped bass (*Morone saxatilis*) and other Bay fisheries (Whipple and co-workers, 1981). While it is clear that substantial quantities of petroleum hydrocarbons are present in the Bay, routine "oil and grease" testing has provided only enough data to raise suspicions that urban runoff may be a significant contributing factor.

Evidence from other areas supports the premise that urban runoff may be a significant contributory factor to hydrocarbon pollution in coastal waters. Connell (1982) found that runoff was the largest source of petroleum hydrocarbon input into the New York Hudson Raritan Estuary (37,000 kg-d⁻¹), while Eganhouse and Kaplan (1982) reported that considerably more petroleum hydrocarbons are introduced from municipal wastewaters than from runoff (about 2-5 times) in the Los Angeles area. These different reports may be indicative of the different climates of the two areas, with much less total runoff coming from Los Angeles than New York City.

The relative contribution of hydrocarbons from sample sites in San Francisco may be expected to fall somewhere between Los Angeles and New York City, since rainfall in the Bay Area is intermediate between those two cities. Further work is expected to establish the relative and absolute magnitude of hydrocarbons from the runoff and municipal wastewater sources in the Bay Area. Investigators looking at the nature and magnitude of hydrocarbons in runoff in other parts of the country have reported similar concentrations (Hoffman and co-workers, 1984; Eganhouse and Kaplan, 1981; Eganhouse and co-workers, 1981; Hunter and co-workers, 1974; Wakeham, 1977; Whipple and Hunter, 1979). Thus, existing evidence indicates that hydrocarbons from runoff can be a significant, if often overlooked, source of chronic environmental pollution.

ANALYTICAL PROCEDURE

The methodology used throughout is outlined in Fig. 1. Water sample size routinely was 4 l. Methylene chloride was chosen as the solvent because of its low boiling point, high density, high dipole moment for extraction of polar organic compounds, and low cost. Comparisons showed it to have nearly identical efficiency to Freon 113, the solvent recommended for "oil and grease" analysis by Standard Methods (APHA, 1985). The extracted organics were separated into a non-aromatic hydrocarbon fraction and aromatic hydrocarbon fraction, a polar fraction, and a very polar fraction by liquid chromatography (deactivated silica gel, 40-140). A 14 cm gel column was sufficient to separate 30 mg of extracted organics. The aliphatic extracts were analyzed using a Varian Vista 6000 chromatograph with SP2100 fused silica column (0.25 mm inner diameter). Initial temperature was 50°C with 4°C rise per min to 280°C and was then isothermal. Injector temperature was 300°C. Helium carrier gas pressure was 245 kPa.

Recoveries for octadecane, anthracene, and 1,2-hydroxystearic acid were from 94 to 99%. Phenol recovery was 89%. GC analysis showed minor carryover of the anthracene into the octadecane fraction, but separation was over 90%. No carryover of the hydroxystearic acid was observed. The lower recoveries of hydroxystearic acid and phenol are attributed to the extraction step, since no loss was detected in the fractionation process.

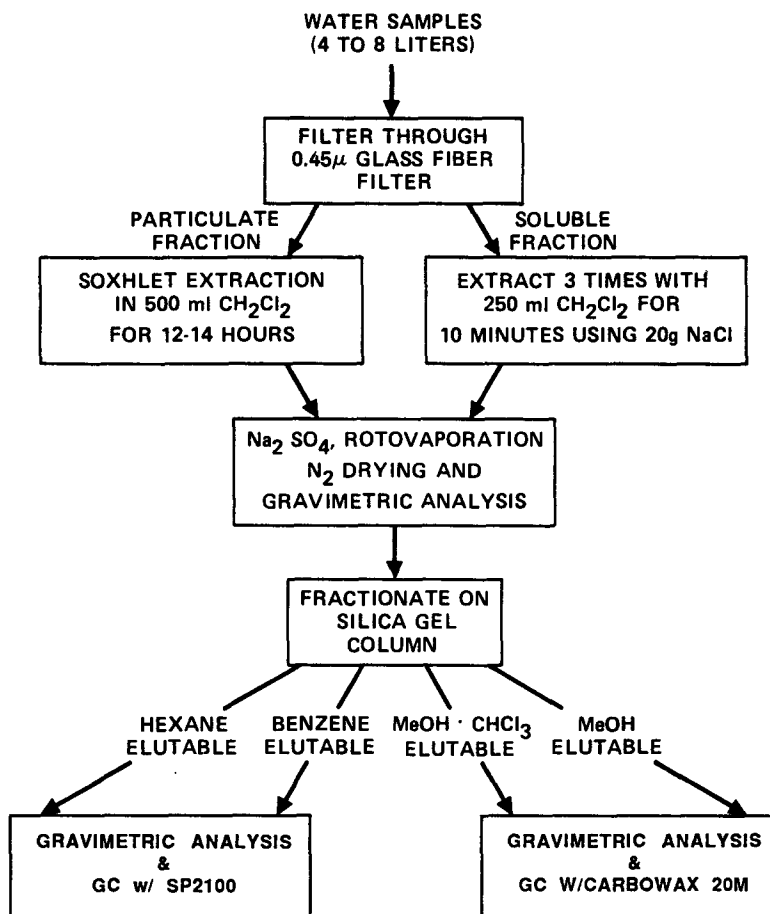


Fig. 1. Analytical protocol for modified "oil and grease" method.

FIELD PROGRAM

The sampling program was conducted at stations selected to represent a diversity of land uses in the San Francisco Bay Area. Land uses for each of the sampled watersheds were determined through an examination of land use maps, census data, and on-site investigation. Watershed boundaries were determined through examination of topographical maps for undeveloped areas, and by using local agency drainage maps for developed regions. The land use distribution of each of the sampled watersheds is shown in Table 1, and their locations are shown on Fig. 2.

In a previous study, "oil and grease" concentrations were examined at several stations within watershed, with samples taken and discharge measured at regular intervals during the active part of a storm (Stenstrom and co-workers, 1984). "Oil and grease" concentration was not significantly related to storm parameters, including time during the storm and time between storms, except for a strong correlation between total "oil and grease" and total rainfall. Similarly, Eganhouse and Kaplan (1981) did not find strong correlations between flow or suspended solids and extracted organic fractions, although Hoffman and co-workers (1982) reported a "first flush" of hydrocarbons at the beginning of a storm. Using this experience, a field program was developed to sample at a maximum number of sites representing different combinations of land uses. While flow characteristics could not be examined, this program allowed substantial evaluation of the relationships between measured parameters representing a large variety of conditions. Of particular interest was the uniformity (or lack thereof) of particular hydrocarbon fractions extracted by the "oil and grease" test as a function of land use.

Table 1. Sampling-site drainage land-use.

		Land Use			Total Area (km ²)	Arterials* (km)
		Residential (%)	Commercial/Industrial (%)	Undeveloped (%)		
1	Richmond	73	22	5	6.47	0.32
2	Sleepy Hollow	26	0	74	8.03	0.0
3	Sonoma	2	0	98	157	0.0
4	Napa	10	3	87	43.8	0.0
5	Temescal	53	13	34	15.3	12.9
6	Glen Echo	51	14	35	2.22	0.0
7	Arroyo Viejo	40	12	48	16.3	2.25
8	Elmhurst	58	34	8	7.77	0.0
9	Pine-Gallinda	23	5	72	68.4	0.0
10	Castro Valley	56	12	32	11.65	5.31
11	Crandall	44	15	41	3.11	2.41
12	Guadalupe	22	4	74	155	12.4
13	Calabazas	56	14	30	37.6	14.6
14	Materdero	5	2	93	7.51	1.93
15	Colma	38	21	41	25.4	12.0

*Freeways and expressways.

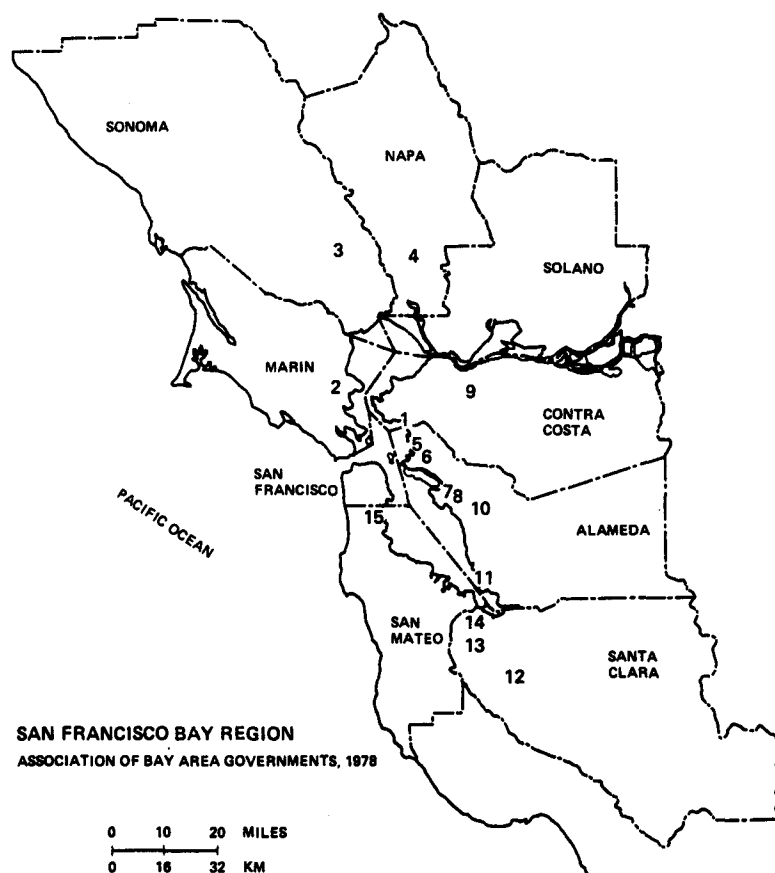


Fig. 2. Study area.

Grab samples were taken at widely scattered stations throughout the San Francisco Bay Area. One sample was taken during each sampling period, and the nature of the flow at the time of sampling was described as: (0) dry weather, (1) at the tail end of the storm hydrograph or following a storm with base flow conditions, (2) following peak flow but with considerable storm flow still in the channel, and (3) during an active rainfall period with considerable flow. Fifteen samples each were taken for individual storms occurring in April, 1984 (rainfall approximately 1.2 cm) and in October, 1984 (rainfall approximately 4.2 cm). Twelve of these stations also were sampled in July, 1984 during dry weather conditions (insufficient water was available in three stations to allow sampling).

Samples were collected either directly in 4 l solvent cleaned glass bottles, or in a stainless steel bucket and quickly transferred to a glass bottle. Samples were taken at turbulent locations, where possible, to ensure a uniform sample. When turbulent zones did not exist, flow was generally sufficiently low to allow sampling of the entire water column with the bucket. These samples were acidified on-site with concentrated hydrochloric acid. Additional samples were taken in 1 l polyethylene bottles for analysis of solids, total organic carbon (TOC) and turbidity. All samples were cooled in ice chests, and kept refrigerated in the laboratory until analyzed.

RESULTS AND DISCUSSION

The results of the three surveys are shown in Tables 2 and 3. Table 2 shows the soluble and particulate extractable materials ("oil and grease") as well as the other measured water quality parameters (total suspended solids or TSS, total dissolved solids or TDS, specified conductivity, pH and turbidity). Table 3 shows the results of the fractionations made on selected samples. Samples were selected from two stations having high commercial land use (Elmhurst and Temescal), three stations having low commercial land use (Guadalupe, Sleepy Hollow and Materdero), and three stations with average commercial land use (Colma, Calabazas, and Crandall).

The data indicate that total "oil and grease" concentration increased dramatically with rainfall. The mean total "oil and grease" concentration for the dry period was $3.03 \text{ mg}\cdot\text{l}^{-1}$ while the mean of the three "wet categories", types 1, 2 and 3, was $8.4 \text{ mg}\cdot\text{l}^{-1}$. Other parameters, including pH, TSS and TDS had different mean values for the wet and dry periods.

The mean aliphatic fraction (mass of aliphatics divided by the total extract mass) was 0.22 and 0.40 for the wet and dry periods, respectively. The difference has questionable statistical significance. A t-test shows significance at the 92% confidence level (assuming equal sample variances); however, the number of samples from each station is not balanced, and there are some data points which contribute very heavily to the result. If one of these is excluded the significance test fails. Further data are required for a definitive result.

The benzene elutable fraction has been termed the aromatic hydrocarbon fraction because it is the fraction in which polynuclear aromatic hydrocarbons will elute. Numerous other compounds (e.g. heavy unsaturated hydrocarbons) will also be collected in this fraction, and we suspect that many plant products (terpenes) are collected in this fraction as well (Nicolas, 1975). This factor may account for the disproportionately high quantities of benzene elutables collected on occasion from the two non-commercial stations may also explain why the hydrocarbon fraction (sum of the aliphatic and aromatic fractions, shown in Table 3) does not vary in any statistically significant way.

An important objective of this work was to determine if land use can be used to predict the significance of organic loading from urban runoff. Correlations were made between various parameters and the land use categories shown in Table 1. Simple Pearson product-moment correlations show correlations between certain land use parameters and water quality parameters. The correlations account for a very small fraction of the variance (typically less than 20%) even though there is less than 5% chance of the correlation being a random occurrence. Commercial land use (percent commercial) and aliphatic fraction were correlated. No correlations existed between arterials (roads) and any other water quality parameter. Eganhouse and co-workers (1981b) were

able to show correlations between storm characteristics and water quality parameters using a similar approach.

To further relate land use and "oil and grease" parameters, an analysis of variance was made, testing aliphatics, aliphatic fraction, total hydrocarbons, and hydrocarbon fraction as a function of commercial land use, using sample type as a class. None of these provided significant results. Therefore, an alternate approach was employed, using quantitative and qualitative data from the chromatograms.

As mentioned previously, the extracts from certain samples were analyzed using high resolution capillary gas chromatography. The chromatograms from the different land use areas show strikingly different aliphatic hydrocarbon distributions. Sleepy Hollow Creek, located in a predominantly non-commercial area, has a bimodal distribution of n-alkanes with a heavy alkane maxima at C₂₉, and has few unresolved peaks, as shown in Fig. 3. In contrast, the chromatogram shown in Fig. 4 from Elmhurst Creek, a station with high commercial land use, has a large unresolved complex mixture (UCM) with a maxima at C₂₃ to C₂₇. This UCM is typical of used motor oil aliphatic fractions such as the one shown in Fig. 5. Motor oils and transmission fluids have very low amounts of n-alkanes (Boyer, 1975; Dell'Acqua, 1975) and it is unlikely that they contribute to the n-alkane envelopes seen in either of the Sleepy Hollow or Materdero chromatograms (not shown). The odd numbered high carbon n-alkane envelopes have generally been attributed to biogenic origins (Eganhouse and co-workers, 1981b; Wakeham, 1977).

Examination of chromatograms from other samples hint that almost every station has a unique aliphatic hydrocarbon pattern. The Colma chromatogram (not shown) contains sharply defined n-alkane peaks in the C₁₆ to C₂₁ range, which are not present in the motor oil chromatogram. Elmhurst Creek, the most commercial station, shows chromatograms which are nearly devoid of n-alkanes. Temescal Creek shows yet another different alkane distribution with a C₁₉ centered unimodal pattern. During our months of sampling the same sampling station sometimes showed different hydrocarbon patterns. Eganhouse and co-workers (1981b) also found variability in hydrocarbon patterns.

Eganhouse and co-workers (1981b) also reported that biogenic sources contribute the odd-numbered hydrocarbons beginning at C₂₅. To test this hypothesis the chromatogram areas for the odd-numbered sequence peaks beginning at C₂₅ were added and compared to the remaining areas. This was done for both soluble and particulate fractions. The percent of C₂₅ and higher peak area of the total peak area ranged from a high of 24.5% for the Materdero particulate aliphatic fraction collected in October, 1984, to 0.9% for the Colma fraction collected in July, 1984. Figure 6 shows the mean of the C₂₅ and higher area portions of the particulate and soluble aliphatic fractions, for the three sampling periods, as a function of percent commercial land use. A statistically significant regression of peak ratios was commercial land use was found, with an r² of 0.6 and an F statistic of 20. The regression was roughly the same for the particulate and soluble fractions, with the same level of significance. The particulate portions were more extreme than the soluble portions, in that higher and lower portions were more apparent for the lowest and highest commercial land uses, respectively.

Similar results were found by Hoffman and co-workers (1983) who measured hydrocarbon discharges from four sampling stations in Rhode Island, reflecting four different land uses, for three to seven rainfall events per station. They used a fractionation technique similar in principle to the one used here. They measured hydrocarbon mass emission rates as a function of total rainfall per event. GC analysis of extracts revealed similar patterns to those shown in Figs. 4 and 5. They found a much higher particulate fraction than found in this investigation. One reason for this difference might be the amount and frequency of rainfall, which was much greater for their study area. More frequent rainfall may prevent buildup of hydrocarbons or particulates.

Table 2. Summary of survey analyses.

Station	Date	Type ¹	Extractables			TSS (mg-l ⁻¹)	TDS (mg-l ⁻¹)	Specific Conductivity (μmhos-cm ⁻¹)	pH	Turbidity (NTU)
			Soluble (mg-l ⁻¹)	Particulate (mg-l ⁻¹)	Total (mg-l ⁻¹)					
Arroyo Viejo	April	3	7.87	11.75	19.62	157	239	230	6.55	45
Arroyo Viejo	Oct	1	1.12	1.83	2.95	44	450	350	8	50
Calabazas	April	2	7.39	6.55	13.94	67	481	295	6.65	32
Calabazas	July	0	1.89	0.75	2.64	2.3	436	600	8.3	2
Calabazas (D)	July	0	2.07	0.85	2.92					
Calabazas	Oct	1	3.59	1.12	4.71	2.3	359	275	7.26	17
Castro Valley	April	3	6.5	16.4	22.9	73	135	125	6.4	31
Castro Valley	July	0	1.28	0.8	2.08	1	648	900	7.85	1.5
Castro Valley	Oct	1	1.86	0.47	2.33	37	650	605	8	50
Colma	April	2	6.11	8.86	14.97	14	1007	800	7.95	13
Colma	July	0	3.98	7.46	11.44	35	1396	1500	8.15	23
Colma	Oct	1	1.5	1.56	3.06	12	1250	900	8.1	19
Colma (D)	Oct	1	1.61	1.57	3.18					
Crandall	April	2	7.91	3.2	11.11	84	1097	1100	7.65	39
Crandall	Oct	1	2.66	1.01	3.67	76	324	300	7.7	50
Elmhurst	April	3	10.11	10.78	20.8	32	135	60	6	15
Elmhurst	July	0	1.6	5.43	7.03	48	359	500	7.45	16
Elmhurst	Oct	1	4.32	4.62	8.94	3	470	400	7.3	12.5
Elmhurst (D)	Oct	1	5.12	5.32	10.44					
Glen Echo	April	3	8.2	5.71	13.9	39	176	110	7	11
Glen Echo	July	0	0.7	0.97	1.67	2.2		3.70	67.6	3
Glen Echo	Oct	1	1.28	1.12	2.4	24	330	400	8.2	9.5
Guadalupe	April	2	10.54	7.21	17.765	105	191	155	6.3	42
Guadalupe	July	0	0.75	0.875	1.62	2	472	550	7.5	13.5
Guadalupe	Oct	1	6.57	3.13	9.7	3	210	125	6.54	41
Materdero	April	2	3.34	4.22	7.56	26	1049	670	7.45	16
Materdero	July	0	0.5	0.625	1.125	1.6	3114	2750	8.2	1
Materdero	Oct	1	1.75	0.58	2.33	6.3	1350	1100	7.9	9
Napa	April	1	0.9	0.75	1.65	109	234	210	7	11
Napa	Oct	1	4.09	3.56	7.65	46	108	45	6.55	28
Napa	July	0	0.62	0.58	1.3	4.4	377	350	7.3	3
Pine-Gallinda	April	2	3.1	1.32	4.42	7	623	750	7.9	3
Pine-Gallinda	April	2	3.55	3.53	7.08	19	145	135	6.55	50
Richmond	April	1	8.442	2.1	10.45	4.6	232	170	6.7	3
Richmond	July	0	1.09	0.622	1.71	1.5	484	490	7.8	1.5
Richmond	Oct	3	4.73	4.37	9.1	32	80	90	6.51	48
Sleepy Hollow	April	1	5.33	3.72	9.05	3.6	289	295	7.4	6
Sleepy Hollow	July	0	1.22	0.79	2.01	12	342	460	7.85	1.5
Sleepy Hollow	Oct	3	1.64	1.99	3.63	433	211	200	7.5	50
Sleepy Hollow (D)	Oct	3	1.95	3.33	5.28					
Sonoma	April	1	1.09	1.33	2.42	23	227	115	7.4	19
Sonoma	Oct	1	0.87	0.36	1.22	28.5	241	290	7.76	14
Sonoma	July	0	0.46	0.477	0.94	1.8	280	335	7.95	1.5
Temescal	April	3	7.26	11.9	19.2	31	151	70	6.4	15
Temescal	July	0	1.29	1.69	2.98	4.3	213	2540	7	4.5
Temescal	Oct	1	1.95	1.85	3.8	3.1	320	350	7.7	4
Temescal (D)	Oct	1	2.39	1.78	4.17					

¹Key: 0 Dry weather; 1 Next day following rainfall; 2 Same day as rain but after peak flow.
3 Active rainfall; (D) Denotes duplicate analysis.

Table 3. Fractionation results.

Station Percent ²	Type ¹	Particulate				Soluble				Percent	
		Aliphatic	Aromatic	Polar	Non- Elutable	Aliphatic	Aromatic	Polar	Non- Elutable	Aliphatic	Aromatic
Calabazas	2	1.910	1.49	1.760	1.39	0.615	0.900	4.54	1.36	18.1	17.1
Colma	0	6.500	0.39	0.630	-0.06	1.870	0.380	1.53	0.20	73.1	6.7
Colma	1	0.560	0.38	0.630	-0.01	0.310	0.110	1.13	-0.05	28.4	25.1
Crandall	2	0.580	0.36	1.545	0.72	0.220	0.170	5.40	2.57	10.5	4.7
Elmhurst	0	2.900	0.88	1.070	0.58	0.250	0.230	0.80	0.37	44.8	15.7
Elmhurst	1	2.750	0.48	1.010	0.38	1.780	0.520	1.72	0.30	50.6	11.2
Guadalupe	2	2.410	1.09	3.700	0.00	1.030	2.440	5.63	1.44	19.4	19.8
Materdero	2	0.535	1.59	0.717	1.38	0.410	0.750	1.74	0.44	12.5	30.9
Materdero	1	0.190	0.19	0.200	0.00	0.090	0.220	1.18	0.26	12.0	17.5
Sleepy Hollow	0	0.180	0.40	0.220	-0.01	0.150	0.290	0.68	0.095	16.4	34.3
Sleepy Hollow	3	0.600	0.24	0.910	0.23	0.110	0.160	0.82	0.550	19.5	11.0
Temescal	0	0.410	0.55	0.730	0.00	0.215	0.215	0.88	-0.020	21.0	25.7
Temescal	1	0.870	0.28	0.710	-0.01	0.310	0.150	1.49	0.000	31.0	11.3

¹Key: 0 Dry weather;
 1 Next day following rainfall;
 2 Same day as rain but after peak flow.
 3 Active rainfall.

²Aliphatic or Aromatic mass (particulate and soluble) as a percent of total extract weight.
 All units are mg·l⁻¹, except fractions (%).

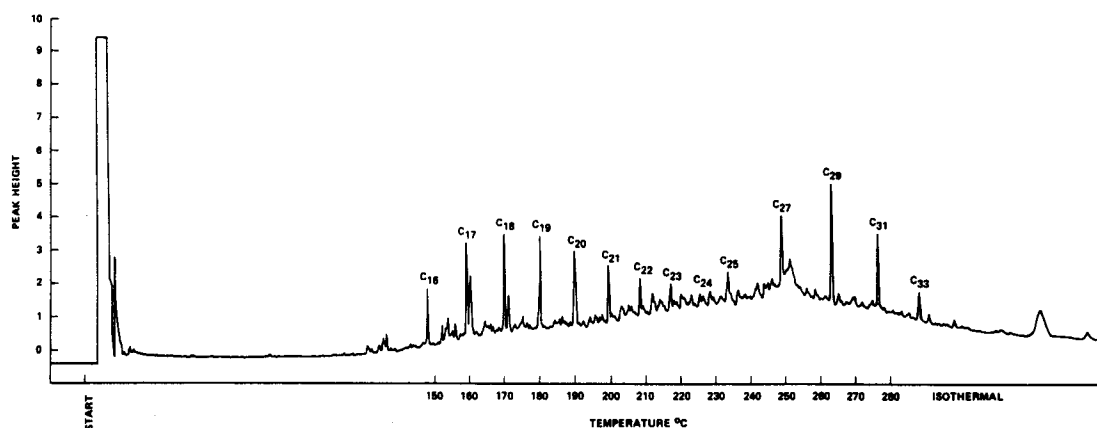


Fig. 3. Chromatogram of aliphatic extract from Sleepy Hollow Creek.

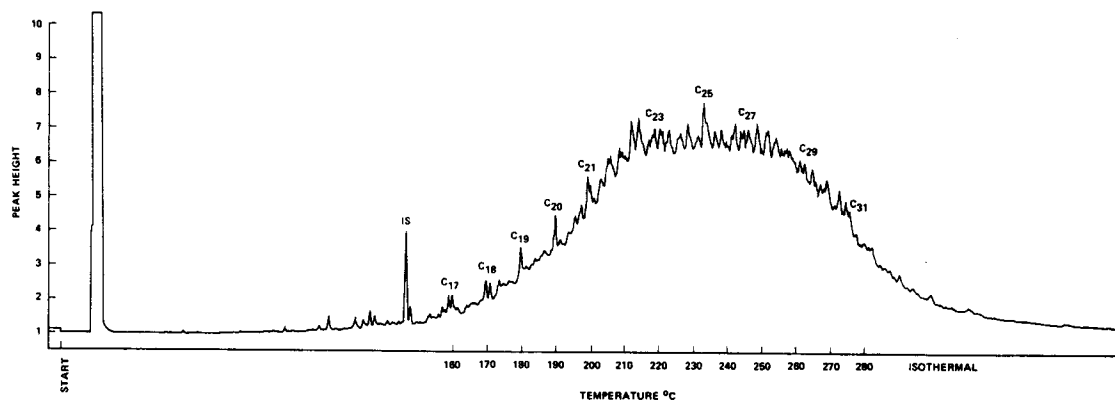


Fig. 4. Chromatogram of aliphatic extract from Elmhurst Creek.

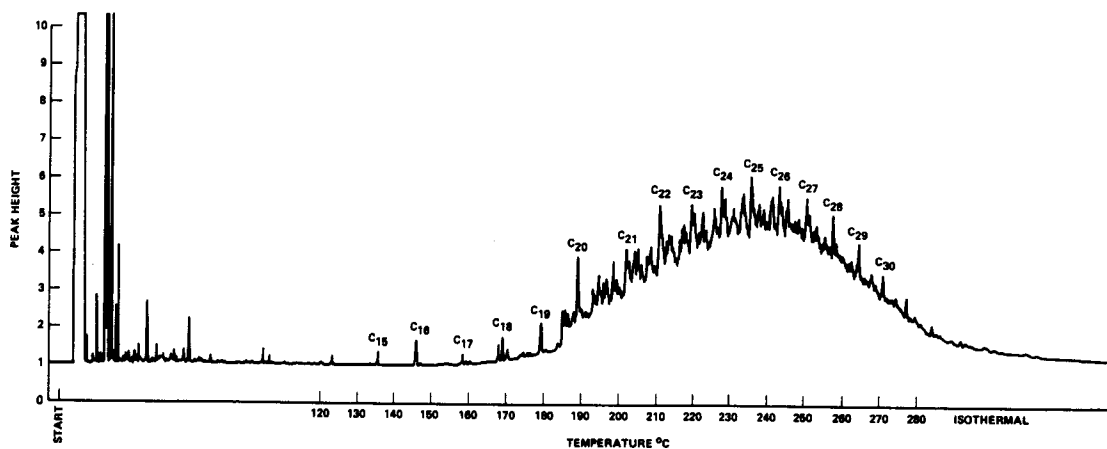


Fig. 5. Chromatogram of aliphatic extract from used motor oil.

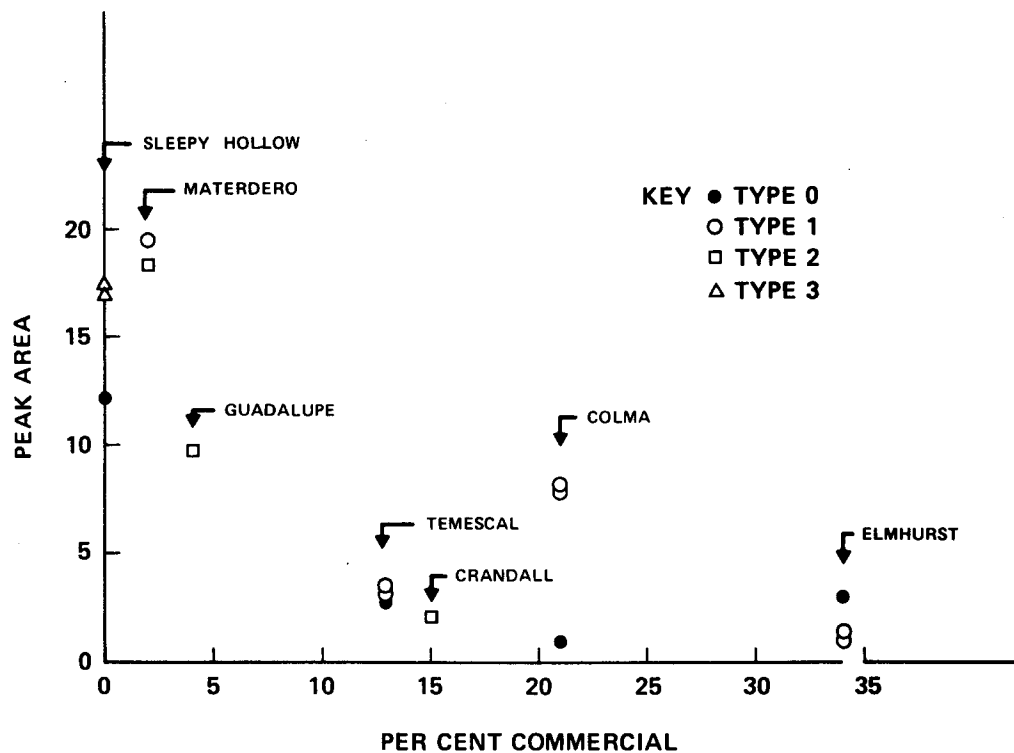


Fig. 6. Percent peak area as a function of commercial land USC.

CONCLUSIONS

Study observations are currently being investigated and we anticipate that examination of chromatograms from the six samples to be collected during this study (only three have been performed to date) will aid in reaching definitive conclusions about aliphatic hydrocarbon distributions and land use. The preliminary results indicate that both biogenic and anthropogenic sources contribute to the hydrocarbons in urban runoff.

The results obtained lead to the following conclusions:

1. Aliphatics show some relation to commercial land use, but the exact relation is not clear. The biogenic contribution appears to mask the relation between aliphatic fraction and commercial land use.
2. The aromatic fraction shows more variability with respect to land use, with some non-commercial stations showing high aromatic fractions.
3. Commercial land use and the concentration of C₂₅ and higher n-alkanes are inversely related. Arterials (roads and freeways) were not related to water quality parameters.

The mean values for many of the water quality parameters were quite different for the wet and dry samplings.

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