

# Fingerprinting BTEX Sources in Groundwater

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## Abstract

Linking the source(s) of BTEX exceedances in hydrocarbon-contaminated groundwater has been shown to be extremely difficult because of differences in partitioning properties of volatile hydrocarbon analytes between potential source oil interfaced with the groundwater. Because aqueous solubility is predicted to be the controlling influence in the partitioning of hydrocarbons from NAPL to groundwater, a solubility-based approach to matching dissolved hydrocarbons in groundwater to their source NAPL has been developed. Aromatic diagnostic hydrocarbons, expressed in multiple pairs, from PIANO (gasoline-range hydrocarbons consisting of paraffin, isoparaffin, (mono-) aromatic, naphthene, and olefin compounds) analysis were identified using analyte aqueous solubility and structure relationships, which were found to be useful in distinguishing the source(s) of hydrocarbons in groundwater. Gasoline and coal tar NAPLs and associated groundwater were chemically characterized at a number of contaminated sites to confirm the diagnostic relationships that can link the BTEX present in remote offsite areas of groundwater to source hydrocarbons at a contaminated site.

Keywords: Hydrocarbon Fingerprinting, PIANO, Mono-Aromatics, Solubility, Coal Tar, Gasoline

## Introduction

Linking hydrocarbons in groundwater to spilled oil or other hydrocarbon-enriched material (e.g., coal tar) is an issue at contaminated sites, especially where there are BTEX (benzene, toluene, ethylbenzene, and xylenes) exceedances from multiple sources that require cleanup and potentially multiple liabilities. BTEX compounds have often been used to determine hydrocarbon sources, but with very limited success. The problem with using BTEX in identifying and differentiating (i.e., 'fingerprinting') sources is that 1) the loss of these highly volatile and soluble hydrocarbons in weathered oil makes correlation of oil and groundwater hydrocarbons difficult; and 2) there are potential changes in BTEX composition in groundwater due to their different physical properties, such as solubility, and microbial degradation preferences. From experiences, the overall composition of oil hydrocarbons (i.e., 'fingerprint') in groundwater does not resemble the 'fingerprint' of the oil (non-aqueous phase liquid - NAPL) in contact with the groundwater. As a result, it is essential to determine sources of hydrocarbons in groundwater to understand the partitioning properties of hydrocarbons of source oil to the groundwater.

The major factors controlling the composition of hydrocarbons in groundwater are the composition of the NAPL and the partitioning behavior of individual constituents of the NAPL<sup>(1)</sup>. The concentrations of NAPL constituents in water interfaced with the NAPL are dependent on the

partitioning equilibrium of the constituents (i.e., solutes) between the aqueous phase and the non-miscible organic phase<sup>(2,3,4)</sup>. Also, concentrations of NAPL constituents in water are controlled kinetically and depend on the rate of molecular diffusion of the solutes in the NAPL phase and at the interface<sup>(4,5,6,7)</sup>. The rate is governed by water temperature, pH, and salinity<sup>(4)</sup>. Constituents in water interfaced with a NAPL may exceed the theoretical solubility due to co-solvency of the other constituents in the NAPL<sup>(4)</sup>.

Because of the different partitioning properties of most hydrocarbons in a NAPL, the relative concentrations of hydrocarbons in the aqueous phase will differ from those originally present in the NAPL. Compared to the hydrocarbon composition in the NAPL, groundwater compositions will have greater concentrations of the more-soluble hydrocarbons, such as BTEX, relative to those of the less-soluble, higher-molecular-weight hydrocarbons such, as C<sub>3</sub>- or C<sub>4</sub>-alkyl benzenes (e.g., 1,2,4-trimethylbenzene) or polycyclic aromatic hydrocarbons (PAHs). Furthermore, many sources share comparable distributions of BTEX compounds. As a result, fingerprinting hydrocarbon sources in groundwater is difficult, especially with a limited set of compounds with different partitioning properties, such as BTEX. Because of the different partitioning properties of the individual BTEX compounds, fingerprinting source NAPL on the basis of only these mono-aromatic hydrocarbons is generally inconclusive.

Because hydrocarbons have different partitioning properties, fingerprinting a NAPL source in groundwater may be enhanced through identifying a set of hydrocarbons that partition similarly from the NAPL to the aqueous phase, thereby maintaining the relative composition of the NAPL (source) in the aqueous phase. The most useful physical property for selecting appropriate volatile hydrocarbons for fingerprinting is solute aqueous solubility<sup>(8)</sup>.

Aqueous solubility of hydrocarbons has been determined to be the controlling influence in the partitioning of hydrocarbons from NAPL to groundwater<sup>(1,9,10)</sup>. Gasoline-range petroleum NAPLs are comprised of many volatile hydrocarbons in addition to BTEX<sup>(9,11)</sup>, which may also be found in contaminated groundwater. Depending on source material, these other volatile hydrocarbons can include paraffins, isoparaffins, alkylated mono-aromatics (aromatics), naphthenes, and olefins (PIANO). Sauer and Costa<sup>(8)</sup> have demonstrated that some of these PIANO compounds, especially the aromatics, are also found in coal tar and coal-tar by-product NAPLs. The aromatic compounds enable the selection of multiple hydrocarbons with similar aqueous solubilities that can be useful in identifying and differentiating different NAPL sources in groundwater.

This paper presents the use of an extensive volatile hydrocarbon characterization (approximately 30 mono-aromatic compounds) and associated diagnostic analyte pairs of hydrocarbon products (NAPLs) and hydrocarbons in groundwater to differentiate sources of volatile hydrocarbons (e.g., BTEX) in groundwater.

## Methods

Groundwater and NAPL were investigated at two sites, one in West Virginia, the other in California. Two wells were sampled at the West Virginia site in June 1998. One well (MW-1) contained LNAPL (gasoline), and the other well (MW-2) without NAPL was located 50 feet down-gradient of MW-1. At the California site, two wells were sampled in May 2000; one well (MW-4) contained the DNAPL (coal tar) and the other well (MW-3) without NAPL was located 105 feet cross-gradient of MW-4.

NAPL and water samples were analyzed for mono-aromatic compounds listed in Table 1 by a purge-and-trap, gas chromatography/mass spectrometry (GC/MS) methodology. The method followed the procedures of EPA Method 8260B (EPA SW-846, 1996) with modifications in chromatographic conditions and target analytes. The purge-and-trap and chromatographic conditions for these analyses were the following: Initial Temperature-35°C, Initial Hold Time-15 min; Initial Ramp Rate-2°C/min to 90°C; Second Ramp Rate-4°C/min to 120°C; Third Ramp Rate-10°C/min to 230°C; Purge Trap Temperature-<35°C; Purge Time-15 min.

Quantification was based on average response factors obtained from a five-point concentration calibration curve. The calibration solutions of target compounds were made up from standard solutions obtained from Supelco Inc. (certified Alphasol PIANO solutions from Air Liquide America Corporation, La Porte, TX). Quality control (QC) samples included continuing calibrations solutions, method blanks, and spiked blanks. Recoveries of all surrogate compounds of the samples were within Method 8260B QC criteria. Reporting limits for target analytes were 10 µg/L for water and 50 mg/kg for NAPL. Woods Hole Group Environmental Laboratories (Raynham, MA) performed the analyses. Although the samples from West Virginia and California were analyzed at different times, the same analytical conditions as prescribed by the standard operating procedures were used for each set of analyses, except that the number of target analytes differed slightly.

## Results

Results of the chemical analyses for the West Virginia and California samples are presented in Table 1. Stacked histograms showing the distributions (fingerprints) of individual aromatic constituents in each sample are displayed in Figure 1 for the West Virginia site samples and Figure 2 for the California site samples. Table 1 also lists solubility values for individual compounds, which were determined either by measurement of specific hydrocarbons in water (Sauer and Costa, 2003).

Although the general aromatic fingerprinting patterns of the NAPLs appeared similar to those of the corresponding groundwater samples (Figures 1 and 2), the relative concentrations of most of the analytes differed between the NAPLs and corresponding groundwater. A closer look shows that only the analytes with similar solubility had consistent relative concentrations in both NAPL and groundwater, e.g., 1,3,5-trimethylbenzene and 1,2,4-trimethylbenzene in MW-1 NAPL and groundwater (Figure 1) and MW-4 NAPL and groundwater (Figure 2). In contrast, constituents with dissimilar solubility (e.g., xylenes and 1,2,4-trimethylbenzene) did not retain their relative concentrations in NAPL and the groundwater in direct contact.

Pairs of analytes (diagnostic ratios) with similar solubility values (ratio  $\sim 1 \pm 0.10$ -0.15) were determined and are listed in Tables 2 and 3 for the West Virginia and California samples, respectively. The ratios of solubility values for the analyte pairings,  $S_w(1)/S_w(2)$ , are shown in column A of the tables. The next three columns (B, C, and D) are the diagnostic ratio values, Concentration(1)/Concentration(2), for the same compounds in NAPL and groundwater samples. Columns E and F provide comparative ratios C/B and C/D, respectively, from columns B, C, and D in the tables.

## Discussion

To determine if the source of hydrocarbons in the groundwater originated from the NAPL at each site, the diagnostic ratios of the groundwater samples were compared to the ratios of the

NAPL. For target analyte pairs of similar solubility (analyte solubility ratio  $\sim 1$ ; column A in Tables 2 and 3), similar diagnostic ratio values for both groundwater and NAPL would indicate that the source of hydrocarbons in the water was from the corresponding NAPL. To facilitate comparison of the NAPL and groundwater diagnostic ratios, comparative ratios were calculated for the NAPL and corresponding groundwater sample taken from the same well. The comparative ratio values for the West Virginia and California samples are shown in column E in Tables 2 and 3, respectively.

Essential to the rationale for selecting analyte pairs of similar solubility as diagnostic tools in source identification is their consistency in both the NAPL and groundwater immediately in contact with the NAPL. The fingerprinting relationship between the NAPL and the groundwater starts at the NAPL-groundwater interface and for this reason groundwater samples were collected at monitoring wells containing the respective NAPLs. Because of the importance of aqueous solubility in soil-groundwater as well as NAPL-groundwater interactions, this approach for selecting diagnostic analyte pairs of similar molecular structure and solubility is expected to provide diagnostic source ratios that are retained downgradient of the source, making the analyte pairs effective tracers of source NAPL. It is reasonable to predict that processes that may alter hydrocarbon distributions based on factors other than solubility, such as selective biodegradation, may affect these ratios, but such factors were not evident for the analyte pairs and the sites included in this study.

As illustrated in Tables 2 and 3, analyte pairs with similar solubility (column A) have groundwater/NAPL ratios of approximately  $1.0 \pm 0.15$ . Similar solubility pairs are grouped in the upper part of the tables. Generally, analyte pairs with solubility differing by 25% are not useful for matching NAPL source with groundwater contamination. The comparative ratios for groundwater/NAPL (column E) in Table 2 that include 1-methyl-2-ethylbenzene (1M2E) were consistently high (approx. 1.3), which may be due to a lesser-than-expected concentration in the NAPL. Conversely, analyte pairs with dissimilar solubility, such as 1,3-dimethyl-5-ethylbenzene/1-methyl-4-ethylbenzene (13M5E/1M4E), have comparative ratio values different from 1.0. Analyte pairs with dissimilar solubility (greater than  $1.0 \pm 15\%$ ) are grouped in the lower part of the tables.

Because of the difficulty of sampling dissolved-phase constituents in groundwater in contact with NAPL, it may be questioned whether the consistency in ratio values from the NAPL to the groundwater in this data set reflected emulsified NAPL in the groundwater. If NAPL emulsions had been included, the diagnostic ratio of almost any pair of target analytes would be the same in the water as in the NAPL. Conversely, if the target analytes were in a soluble form, then target analyte pairs with dissimilar solubility values would show different ratios in water compared to the NAPL, while target analytes of similar solubility values would retain ratios in water compared to the NAPL. The diagnostic ratios of analytes with dissimilar solubility values (e.g., 1,3-dimethyl-5-ethylbenzene/1-methyl-4-ethylbenzene [13M5E/1M4E] and 1,2-dimethyl-4-ethylbenzene/1-methyl-2-ethylbenzene [12M4E/1M2E]) were calculated (Tables 2 and 3) and illustrate the predicted differences for NAPL and groundwater from analytes of dissimilar solubility.

Groundwater samples collected a distance away from the NAPL source at the West Virginia and California sites were analyzed for target analytes to see if the hydrocarbons in these remote groundwater samples originated from the respective NAPLs. Comparative ratio values were calculated for analyte pairs from the remote groundwater sample and the source groundwater sample for the two sites (column F in Tables 2 and 3). At the California site (Table 3), diagnostic ratios of the cross-gradient groundwater sample (MW-3) were different than those of the NAPL and groundwater at MW-4, indicating that the remote groundwater hydrocarbons did not originate from the coal tar NAPL. At the West Virginia site (Table 2), the diagnostic ratios of the downgradient

groundwater sample (MW-2, column D) were found to be very similar to those of the NAPL and groundwater from MW-1 (columns B and C) indicating the source of hydrocarbons to groundwater in MW-2 was the gasoline NAPL in MW-1. The comparable diagnostic ratios of the MW-1 groundwater (Table 2, column C) and the remote MW-2 groundwater (column D) provide evidence that the selected ratios were retained during transport in the aquifer.

An interesting observation regarding the groundwater samples at the West Virginia site was that the diagnostic ratio values of analyte pairs with dissimilar solubilities (e.g., 1,3-dimethyl-5-ethylbenzene/1-methyl-4-ethylbenzene [13M5E/1M4E]) were similar (comparative ratio ~ 1.0, column F). An interpretation of this empirical observation is that once in the groundwater, the relative distributions of many target analytes are retained during transport. However, changes during transport, especially over longer distances than evaluated in this study (approximately 50 ft), may be expected for analytes of dissimilar properties that would influence analyte transport (e.g., preferential degradation or adsorption to organic material and soil particles). Although some analyte pairs with dissimilar solubilities may be useful in fingerprinting hydrocarbons at different groundwater locations, these pairs would not be useful for attributing volatile hydrocarbons in groundwater to their NAPL source.

## Conclusion

Through the application of a solubility-based approach, this study demonstrates that analyzing for the alkyl benzenes with 3, 4, and 5 carbon groups (C<sub>3</sub>, C<sub>4</sub>, and C<sub>5</sub>-alkyl benzenes) present in fuel oils and other hydrocarbon products and pairing analytes with similar solubilities provides conclusive source diagnostic fingerprints for identifying and differentiating sources of volatile hydrocarbon (including BTEX) in groundwater. The diagnostic ratios that are most useful, but not limited to, are:

- 1-Methyl-3-ethylbenzene/1-Methyl-4-ethylbenzene
- 1,3,5-Trimethylbenzene/1,2,4-Trimethylbenzene
- 1-Methyl-3-propylbenzene/1-Methyl-4-propylbenzene
- 1,4-Dimethyl-2-ethylbenzene/1,2-Dimethyl-4-ethylbenzene
- 1-Methyl-2-ethylbenzene/1,2,3-Trimethylbenzene
- 1-Methyl-2-propylbenzene/ 1,2-Dimethyl-4-ethylbenzene

Whereas geochemical conditions did not appear to influence the solubility dynamics, it is prudent to select several diagnostic pairs when applying this solubility-based fingerprinting model.

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**Table 1.** Alkyl Benzene Results for NAPL and Groundwater (GW) Samples from West Virginia (WV) and California (CA) Sites

	Abbv.	S <sub>w</sub> <sup>1</sup> (@ 25°C) mg/L	MW-1 NAPL	MW-1	MW-2	MW-4 NAPL	MW-4	MW-3
			WV	WV	WV	CA	CA	CA
			LNAPL mg/kg oil	GW ug/L	GW ug/L	DNAPL mg/kg oil	GW ug/L	GW ug/L
Benzene	B	1790	ND	601	2,620	ND	48.2	ND
Toluene	T	535	7,800	3,230	7,100	97.5	289	2.3
Ethylbenzene	E	161	13,700	2,020	1,460	790	653	92.0
m/p-Xylenes	mpX	151	72,000	15,700	11,500	2,270	1,692	48.6
o-Xylene	oX	171	29,500	8,710	5,930	1,420	931	2.04
Isopropylbenzene	IP	65	2,400	177	104	270	78.4	44.9
n-Propylbenzene	NP	51	10,900	378	165	111	22.8	158
1-Methyl-3-ethylbenzene	1M3E	83	71,000	7,520	4,290	1,500	351	32.2
1-Methyl-4-ethylbenzene	1M4E	93	15,800	1,690	1,270	769	154	44.3
1,3,5-Trimethylbenzene	135M	48	29,100	3,100	2,190	1,080	270	42.2
1-Methyl-2-ethylbenzene	1M2E	74	19,200	2,700	1,990	478	104	51.4
1,2,4-Trimethylbenzene	124M	52	98,000	9,820	6,430	3,710	908	126
1,2,3-Trimethylbenzene	123M	75	NA	NA	NA	3,950	783	371
Isobutylbenzene	IB	10	1,400	48.7	15.2	ND	ND	10.4
sec-Butylbenzene	SB	18	1,200	41.5	18.2	ND	ND	13.1
1-Methyl-2-isopropylbenzene	1M2I	48	15,700	678	452	NA	NA	NA
1-Methyl-3-isopropylbenzene	1M3I	43	2,370	125	79	304	31.2	9.0
1-Methyl-4-isopropylbenzene	1M4I	23	NA	NA	NA	397	38.0	6.3
1-Methyl-3-propylbenzene	1M3P	9.1	4,350	162	89.3	145	9.9	17.2
1-Methyl-4-propylbenzene	1M4P	10	8,350	311	154	114	8.2	45.7
n-Butylbenzene	NB	12	4,150	79.4	ND	64.5	3.4	28.6
1,3-Dimethyl-5-ethylbenzene	13M5E	12	18,800	811	545	507	39.3	20.1
1,2-Diethylbenzene	12E	23	1,290	79.6	55.8	32.7	2.9	16.1
1,3-Diethylbenzene	13E	27	NA	NA	NA	ND	ND	180
1-Methyl-2-propylbenzene	1M2P	13	6,110	286	181	67.5	5.4	58.0
1,4-Dimethyl-2-ethylbenzene	14M2E	15	13,000	692	417	302	26.5	49.5
1,2-Dimethyl-4-ethylbenzene	12M4E	13	21,600	1,060	680	640	54.2	141
1,3-Dimethyl-2-ethylbenzene	13M2E	20	NA	NA	NA	71.8	7.3	21.5
1,2-Dimethyl-3-ethylbenzene	12M3E	17	NA	NA	NA	159	14.3	28.6
1,2,4,5-Tetramethylbenzene	1245M	3.5	NA	NA	NA	524	41.5	137
2-Methylbutylbenzene	2MB	10	NA	NA	NA	ND	ND	1.4
Pentylbenzene	PEB	3.4	2,070	ND	ND	43.0	ND	ND

ND-Not detected; NA-Not analyzed

1 – Solubility values referenced in Sauer and Costa (2003)<sup>(8)</sup>

**Table 2.** Diagnostic and Comparative Ratios of Gasoline NAPL and Associated Groundwater (GW) Samples

Analyte Pairs	Diagnostic Ratios				Comparative Ratios	
	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>
	Solubility Ratios	MW-1 NAPL	MW-1 GW	MW-2 GW	MW-1 GW/ MW-1 NAPL	MW-1 GW/ MW-2 GW
135M/124M	0.92	0.30	0.32	0.34	1.07	0.93
1M3E/1M4E	0.89	4.50	4.46	3.37	0.99	1.32
1M3P/1M4P	0.91	0.52	0.52	0.58	1.00	0.90
14M2E/12M4E	1.15	0.60	0.65	0.61	1.09	1.07
1M2E/1M4E	0.80	1.22	1.60	1.56	1.31	1.02
1M2E/123M	0.99	NA	NA	NA	--	--
1M2P/12M4E	1.00	0.28	0.27	0.27	0.96	1.02
1M2E/1M3E	0.89	0.27	0.36	0.46	1.33	0.77
123M/124M	1.27	NA	NA	NA	--	--
1M2E/124M	1.42	0.20	0.27	0.31	1.40	0.89
13M5E/1M4E	0.13	1.19	0.48	0.43	0.40	1.12
12M4E/1M2E	0.18	1.13	0.39	0.34	0.35	1.15

NA = not analyzed

**Table 3.** Diagnostic and Comparative Ratios of Coal Tar NAPL and Associated Groundwater (GW) Samples

Analyte Pairs	<u>Diagnostic Ratios</u>				<u>Comparative Ratios</u>	
	<u>A</u> Solubility Ratios	<u>B</u> MW-4 NAPL	<u>C</u> MW-4 GW	<u>D</u> MW-3 GW	<u>E</u> MW-4 GW/ MW-4 NAPL	<u>F</u> MW-4 GW/ MW-3 GW
135M/124M	0.92	0.29	0.30	0.33	1.02	0.89
1M3E/1M4E	0.89	1.94	2.27	0.73	1.17	3.12
1M3P/1M4P	0.91	1.27	1.21	0.38	0.96	3.21
14M2E/12M4E	1.15	0.47	0.49	0.35	1.04	1.39
1M2E/1M4E	0.80	0.62	0.67	1.16	1.08	0.58
1M2E/123M	0.99	0.12	0.13	0.14	1.10	0.96
1M2P/12M4E	1.00	0.11	0.10	0.41	0.95	0.24
1M2E/1M3E	0.89	0.32	0.30	1.59	0.93	0.19
123M/124M	1.27	1.07	0.86	2.94	0.81	0.29
1M2E/124M	1.42	0.14	0.11	0.41	0.78	0.34
13M5E/1M4E	0.13	0.66	0.25	0.45	0.38	0.56
12M4E/1M2E	0.18	1.34	0.52	2.74	0.39	0.19

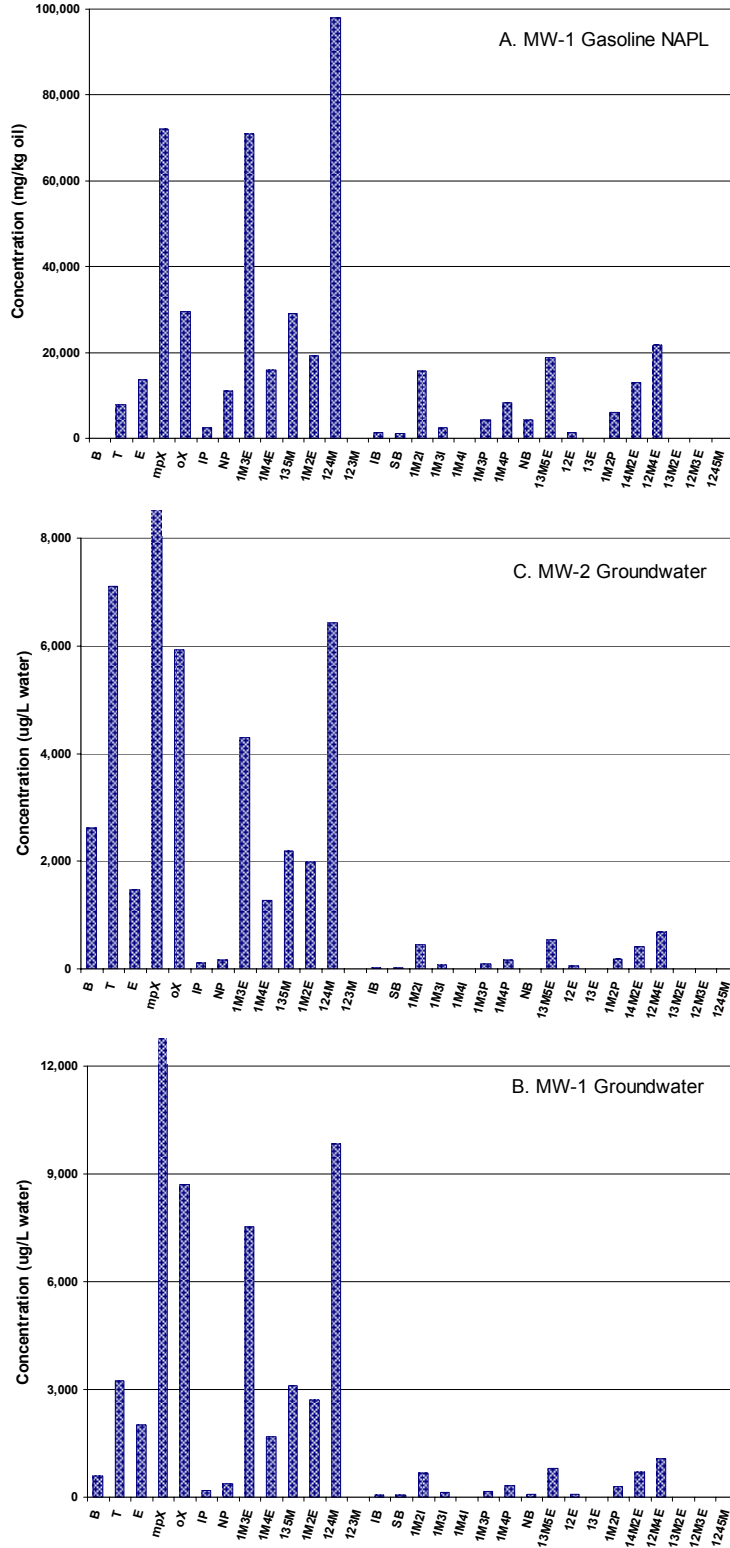
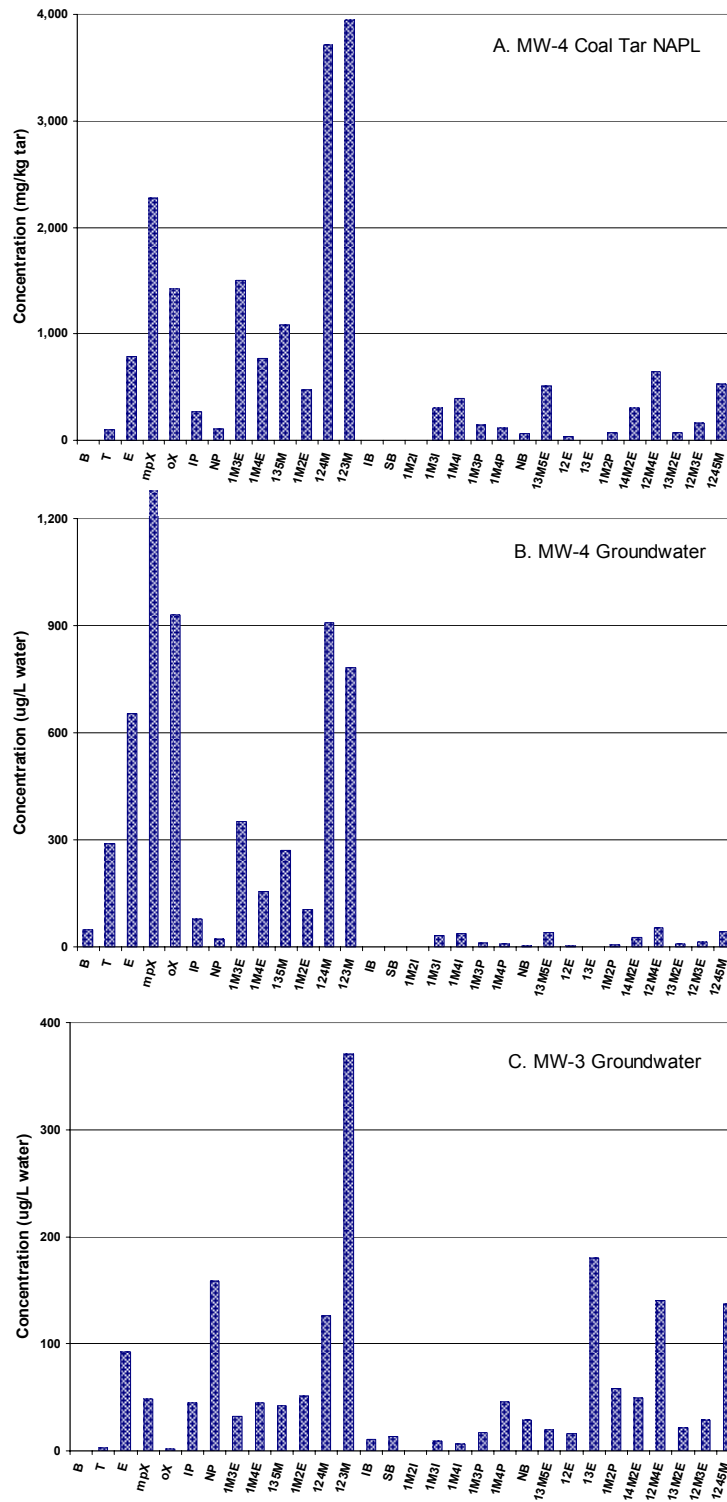


Figure 1. Alkyl benzene distributions in NAPL and groundwater from the West Virginia site.



**Figure 2.** Alkyl benzene distributions in NAPL and groundwater from the California site