



December 1, 2012

Roya C. Kambin
Project Manager
Marketing Business Unit

**Chevron Environmental
Management Company**
6101 Bollinger Canyon Road
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Mr. Jerry Wickham
Alameda County Health Care Services
1131 Harbor Bay Parkway, Suite 250
Alameda, California 94502

RE: Compound Specific Isotope Analysis Report
1771 First Street, Livermore, California
Fuel Leak Case No.: RO0000436

RECEIVED
3:38 pm, Dec 03, 2012
Alameda County
Environmental Health

Dear Mr. Wickham,

I declare under penalty of perjury that to the best of my knowledge the information and/or recommendations contained in the attached report is/are true and correct.

If you have any questions or need additional information, please contact me at (925) 790-6270.

Sincerely,

A handwritten signature in black ink, appearing to read "Roya Kambin".

Roya Kambin
Union Oil of California – Project Manager

Attachment
Compound Specific Isotope Analysis Report



Mr. Jerry Wickham
Alameda County Health Care Services Agency
1131 Harbor Bay Parkway, Suite 250
Alameda, California 94502

ARCADIS U.S., Inc.
2000 Powell Street
Suite 700
Emeryville
California 94608
Tel 510 652 4500
Fax 510 652 4906
www.arcadis-us.com

Subject:

Compound Specific Isotope Analysis for former Unocal #4186 located at 1771 First Street, Livermore, CA

Dear Mr. Wickham:

On behalf of Chevron Environmental Management Company, for itself and as Attorney-in-Fact for Union Oil Company of California, ARCADIS U.S., Inc is pleased to submit this report documenting the results of a Compound Specific Isotope Analysis (CSIA) proposed in the February 9, 2011 Conestoga-Rovers & Associates (CRA) "Supplemental Monitoring and Sampling Work Plan" approved by Alameda County Environmental Health (ACEH) in a letter dated March 28, 2011.

In June 2011, TRC Solutions at the direction of CRA collected groundwater samples from five monitoring wells (U-3, U-5, U-9, U-10, and U-11), Figure 1, for analysis of the stable carbon and hydrogen isotopic composition of methyl tertiary-butyl ether (MTBE) and stable carbon isotopic composition of tertiary-butyl alcohol (TBA). The compound-specific CSIA were conducted to improve the understanding of MTBE biodegradation at the site and to provide a line of evidence supporting degradation of MTBE as the primary source for concentrations of dissolved TBA.

Monitoring well U-3 is screened in the shallow groundwater zone near the source area; the other monitoring wells (U-5, U-9, U-10, and U-11) are screened in the intermediate groundwater zone, with U-10 in the source area, U-9 downgradient of the source area, and U-5 and U-11 cross-gradient of the source area. Groundwater samples were shipped to the University of Oklahoma for the carbon and hydrogen isotopic analyses of MTBE ($\delta^{13}\text{C}$ and $\delta^2\text{H}$) and TBA ($\delta^{13}\text{C}$).

Background on MTBE/TBA Isotopes

Isotopes are atoms of the same element that have different masses due to differing numbers of neutrons in the nucleus. For example, hydrogen atoms can contain nuclei with no neutrons (hydrogen, ^1H), one neutron (deuterium, ^2H or D), or two neutrons (tritium, ^3H). Stable isotopes remain unchanged while radioactive isotopes, such as carbon-14 (^{14}C), decay to stable isotopes. Although stable isotopes

Imagine the result

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Our ref:
B0047942.2012

inherently remain unchanged, the amount (composition) of a particular stable isotope within a specific compound changes (i.e., isotopic fractionation occurs) when that compound is degraded. Molecules of compounds containing isotopes of lighter mass (e.g., ^1H , ^{12}C) are preferentially degraded, causing a shift in the isotopic composition of the overall pool of that particular compound. Biological and/or chemical degradation processes (e.g., biological oxidation, biological reduction, abiotic degradation, in-situ chemical oxidation, and in-situ chemical reduction) can cause significant isotopic fractionation. Conversely, non-degradation processes such as sorption, volatilization, and dilution typically have a negligible effect on the composition of stable isotopes. Therefore, CSIA data can be used to determine whether degradation is occurring and, when evaluated in concert with site biogeochemical data, which mechanism is primarily responsible for the degradation (e.g., aerobic biodegradation, anaerobic biodegradation).

The convention for reporting isotopic ratios is the delta (δ) notation, which uses units of per mil (‰) because the values are usually less than 0.05. The delta notations for $\delta^{13}\text{C}$ and $\delta^2\text{H}$, or δD , are defined by the following equations, which normalize the isotope ratio of the sample to the isotope ratio of a known standard:

$$\delta^{13}\text{C} = \left[\frac{(^{13}\text{C}/^{12}\text{C})_{\text{sample}}}{(^{13}\text{C}/^{12}\text{C})_{\text{standard}}} - 1 \right] * 1000$$

$$\delta\text{D} = \left[\frac{(\text{D}/^1\text{H})_{\text{sample}}}{(\text{D}/^1\text{H})_{\text{standard}}} - 1 \right] * 1000$$

The international stable carbon isotope standard is Vienna Pee Dee Belemnite, and the international stable hydrogen isotope standard is Vienna Standard Mean Ocean Water (Clark and Fritz 1997).

Degradation of MTBE and its daughter product TBA can occur via chemical or biological oxidation (via naturally occurring aerobic and/or anaerobic microorganisms). During MTBE degradation to TBA, MTBE molecules with ^{12}C at the reaction site are metabolized more rapidly than MTBE molecules with ^{13}C . As a result, the $^{13}\text{C}/^{12}\text{C}$ ratio of the residual pool of MTBE is enriched in the heavier isotope and the $\delta^{13}\text{C}$ value of the residual MTBE increases (becomes more positive). Similarly, enrichment of the hydrogen isotope of MTBE and enrichment of the carbon isotope of TBA can be observed during degradation of each compound, increasing δD of MTBE and $\delta^{13}\text{C}$ of TBA, respectively. Therefore, analysis of select site groundwater samples for isotopic signature of MTBE and TBA can provide a means for confirming in-situ degradation of site constituents of concern.

Results

Concentration Data – Methyl Tertiary-Butyl Ether and Tertiary-Butyl Alcohol

Table 1 (below) includes MTBE and TBA concentration data and TBA/MTBE ratios. Figures 2 through 6 provide concentration vs. time plots for MTBE and TBA for all the wells. The historical data suggest that MTBE is degrading to TBA in U-3, U-9, U-10, and U-11 because the TBA levels are high and seem to track (or follow) the MTBE levels. The TBA/MTBE ratios computed in Table 2 indicate MTBE biodegradation and the formation of TBA in groundwater at four of the five wells (U-3, U-9, U-10 and U-11) because the TBA/MTBE values are greater than 1. Well U-5 appears to have relatively low concentrations of both MTBE and TBA and an MTBE/TBA ratio significantly less than 1.

Table 1. MTBE and TBA Results – June 2011

Well	Position	MTBE (µg/L)	TBA (µg/L)	TBA/MTBE Ratio
U-3	Source	39	9,600	246.2
U-5	Crossgradient	55	9.9	0.18
U-9	Downgradient	65	110	1.7
U-10	Source	350	2,900	8.3
U-11	Crossgradient	3,600	6,500	1.8
Notes: µg/L – micrograms per liter				

Groundwater Reduction-Oxidation Condition

Degradation of petroleum hydrocarbons, including MTBE, in groundwater can proceed via aerobic or anaerobic microbial processes. Bacteria present in soil and groundwater obtain energy for cell production and maintenance by facilitating thermodynamically advantageous oxidation-reduction reactions involving the transfer of electrons from electron donors to available electron acceptors. When sufficient dissolved oxygen is present in groundwater, biodegradation of MTBE and other hydrocarbons proceeds aerobically (with oxygen as the electron acceptor). As oxygen becomes less available, anaerobic microorganisms consume electron acceptors in the following order of preference: nitrate, manganese oxides, ferric iron hydroxides/oxyhydroxides, sulfate, and carbon dioxide. Anaerobic biodegradation

processes are thus associated with decreased concentrations of nitrate and sulfate, increased concentrations of dissolved ferrous iron, and production of methane within the plume as compared with background (Wiedemeier et al. 1999).

Groundwater geochemistry data provided in Table 2 (below) show that strongly reducing conditions are present in the source (U-3 and U-10) and downgradient (U-9) areas, while mildly reducing to aerobic conditions are present in the crossgradient area (U-5 and U-11). The interpretation of strongly reducing conditions at U-9 and U-10 is supported by concentrations of nitrate below laboratory reporting limits, low sulfate concentrations relevant to crossgradient (U-5 and U-11) locations, and detected concentrations of ferrous iron and methane. Moderately reducing conditions are indicated for groundwater at crossgradient well U-11 based on nitrate concentrations below laboratory reporting limit, lower ferrous iron and methane concentrations, and higher sulfate concentration than at U-9 and U-10. Groundwater geochemistry data for cross-gradient well U-5 suggest aerobic to mildly reducing conditions based on the detected concentration of nitrate, no detection of ferrous iron, and low detected concentration of methane.

Table 2. Redox Data – June 2011

Well	Position	Nitrate (mg/L)	Sulfate (mg/L)	Ferrous iron (mg/L)	Methane (mg/L)	Redox Condition
U-3	Source	<0.44	<1.0	3,300	1.4	Strongly reducing/anaerobic
U-5	Cross-gradient	3.5	33	<100	0.0065	Oxic/aerobic
U-9	Downgradient	<0.44	10	470	4.6	Strongly reducing/anaerobic
U-10	Source	<0.44	3.4	930	5.6	Strongly reducing/anaerobic
U-11	Cross-gradient	<0.88	1000	140	0.64	Moderately reducing/anaerobic
Notes: mg/L – milligrams per liter						

C and H isotopes – Methyl Tertiary-Butyl Ether and Tertiary-Butyl Alcohol

On Figure 7, carbon ($\delta^{13}\text{C}$) versus hydrogen ($\delta^2\text{H}$) isotope values for MTBE are plotted for the five monitoring wells. The $\delta^{13}\text{C}$ data range for TBA observed at these wells is also plotted alongside the range of $\delta^{13}\text{C}$ and $\delta^2\text{H}$ data (solid orange box), including the range for analytical precision (green-dashed-line box) observed for

undegraded MTBE. Any data observed to be outside of the green-dashed-line box is significantly different from the expected starting composition and has been degraded either aerobically (red arrow/purple zone trajectory) or anaerobically (blue arrow/zone trajectory).

Methyl Tertiary-Butyl Ether

Reported values of $\delta^{13}\text{C}$ of MTBE ranged from -28.7‰ (U-5) to $+9.5\text{‰}$ (U-3). Reported values of $\delta^2\text{H}$ of MTBE ranged from -71‰ (U-5) to -11‰ (U-3). The laboratory analytical precision for $\delta^{13}\text{C}$ of MTBE and TBA is 0.5‰ ; therefore, differences in $\delta^{13}\text{C}$ values greater than 1‰ are considered significant. The laboratory analytical precision for $\delta^2\text{H}$ of MTBE is 3‰ ; therefore, differences in $\delta^2\text{H}$ values greater than 6‰ are considered significant.

As presented above, biological degradation of MTBE will result in a shift (or enrichment) in $\delta^{13}\text{C}$ and $\delta^2\text{H}$ toward more positive values. The expected starting (or "benchmark") composition for undegraded MTBE in gasoline ranges from approximately -27.4 to -33‰ for $\delta^{13}\text{C}$ and from -80 to -125 for $\delta^2\text{H}$ (Kuder et al. 2005) (Figure 7). Based on analytical precision for stable carbon and hydrogen isotope analyses, $\delta^{13}\text{C}$ values higher (more positive) than approximately -26.4‰ and $\delta^2\text{H}$ values higher than approximately -74‰ may indicate that biological degradation of MTBE is occurring.

Carbon is enriched above the benchmark range for undegraded MTBE by greater than 35‰ in the U-3 groundwater sample and by approximately 1‰ in groundwater at U-9 and U-10. These results indicate that biological degradation of MTBE is occurring in groundwater near U-3 and may be occurring in groundwater near U-9 and U-10. The $\delta^2\text{H}$ values for all groundwater samples are enriched relative to the expected benchmark $\delta^2\text{H}$ composition of MTBE, with the largest isotopic shift (69‰) measured for MTBE in the sample from U-3. These results also indicate that degradation of MTBE is likely occurring.

The magnitude in the shift of $\delta^{13}\text{C}$ and $\delta^2\text{H}$ values can be used to assess potential biodegradation pathways. For example, as shown on Figure 7, degradation of MTBE under aerobic conditions will result in relatively small shifts in $\delta^{13}\text{C}$ values and relatively large shifts in $\delta^2\text{H}$ values (Gray et al. 2002, Rosell et al. 2007, McKelvie et al. 2009, Hunkeler et al. 2001, Lesser et al. 2008, Youngster et al. 2010, Bastida et al. 2010, Jechalke et al. 2011), while degradation of MTBE under anaerobic conditions will result in relatively large shifts in $\delta^{13}\text{C}$ values and moderate shifts in $\delta^2\text{H}$

values (Kolhatkar et al. 2002, Kuder et al. 2002, Kuder et al. 2005, Somsamek et al. 2005, Zwank et al. 2005, Somsamek et al. 2006, Youngster et al. 2010).

Based on this characterization of expected stable carbon and hydrogen isotopic composition shifts, $\delta^{13}\text{C}$ and $\delta^2\text{H}$ values for MTBE in site groundwater samples suggest that MTBE is being degraded under both aerobic and anaerobic pathways. However, groundwater geochemical data and the accumulation of TBA suggest that MTBE degradation is occurring primarily via anaerobic degradation. TBA will degrade readily under aerobic conditions, but will tend to accumulate under anaerobic conditions. Regardless of the specific degradation pathway, the stable carbon and hydrogen isotopic composition of MTBE in groundwater demonstrate that MTBE is undergoing biological degradation.

Tertiary-Butyl Alcohol

Reported values of $\delta^{13}\text{C}$ of TBA ranged from -27.3‰ (U-11) to -26.1‰ (U-10). Because concentrations of TBA in U-5 were below the laboratory detection limits for CSIA, $\delta^{13}\text{C}$ results were not available for U-5. As a result, the data set for $\delta^{13}\text{C}$ TBA contains only four data points (U-3, U-9, U-10, and U-11). The carbon signatures of TBA observed in monitoring wells U-9, U-10, and U-11 overlaps with the carbon signatures of MTBE at those same wells. This suggests that the TBA present at these wells is likely the daughter product of aerobic MTBE degradation in these wells and that the system may resemble a mature closed system, which restrains the isotopic composition difference between products and reactants. The TBA in these wells is likely formed and rapidly degraded. Conversely, the TBA formed near U-3 is likely formed through anaerobic MTBE degradation (which causes the enriched $\delta^{13}\text{C}$ MTBE value of 9.5‰ observed), but TBA accumulates due to a lack of significant degradation, thereby not showing a fractionation effect related to the enriched MTBE at that well. In line with this conceptual model, the TBA concentration is highest and the MTBE concentration is lowest at this location, further supporting the lack of degradation/accumulation theory.

Conclusions

Based on the CSIA results, TBA/MTBE ratios, and biogeochemical data, degradation of MTBE is occurring in site groundwater. This conclusion is based on the following lines of evidence:

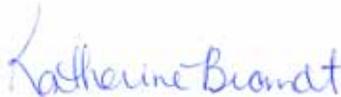
- MTBE concentrations are stable to decreasing across the site.

- TBA concentrations are generally higher than MTBE concentrations, indicating degradation of MTBE to TBA.
- The ratio of TBA to MTBE is increasing at most locations, indicating ongoing degradation of MTBE in site groundwater. The relatively high TBA/MTBE ratios suggest a mature plume that will continue to attenuate with time.
- The stable carbon and hydrogen isotopic composition of MTBE demonstrates enrichment above the expected starting, or benchmark, composition of MTBE in gasoline. The stable carbon isotopic composition indicates that TBA is derived from degradation of MTBE.

If you have any questions or comments regarding the contents of this letter, please contact Ms. Roya Kambin of Chevron at 925.790.6270 or by e-mail at RKambin@Chevron.com. Alternatively, you may contact Katherine Brandt of ARCADIS at 510.596.9675 or by e-mail at Katherine.Brandt@arcadis-us.com.

Sincerely,

ARCADIS U.S., Inc.



Katherine Brandt
Certified Project Manager



David W. Lay, P.G., C.P.G.
Principal Geologist



Copies:

Roya Kambin, CEMC (electronic copy)

Eric Hetrick, ConocoPhillips (electronic copy)

Thomas T. and Celine T. Vadakkekunnel, 481 Peacock Court, Dublin, CA 94568

References

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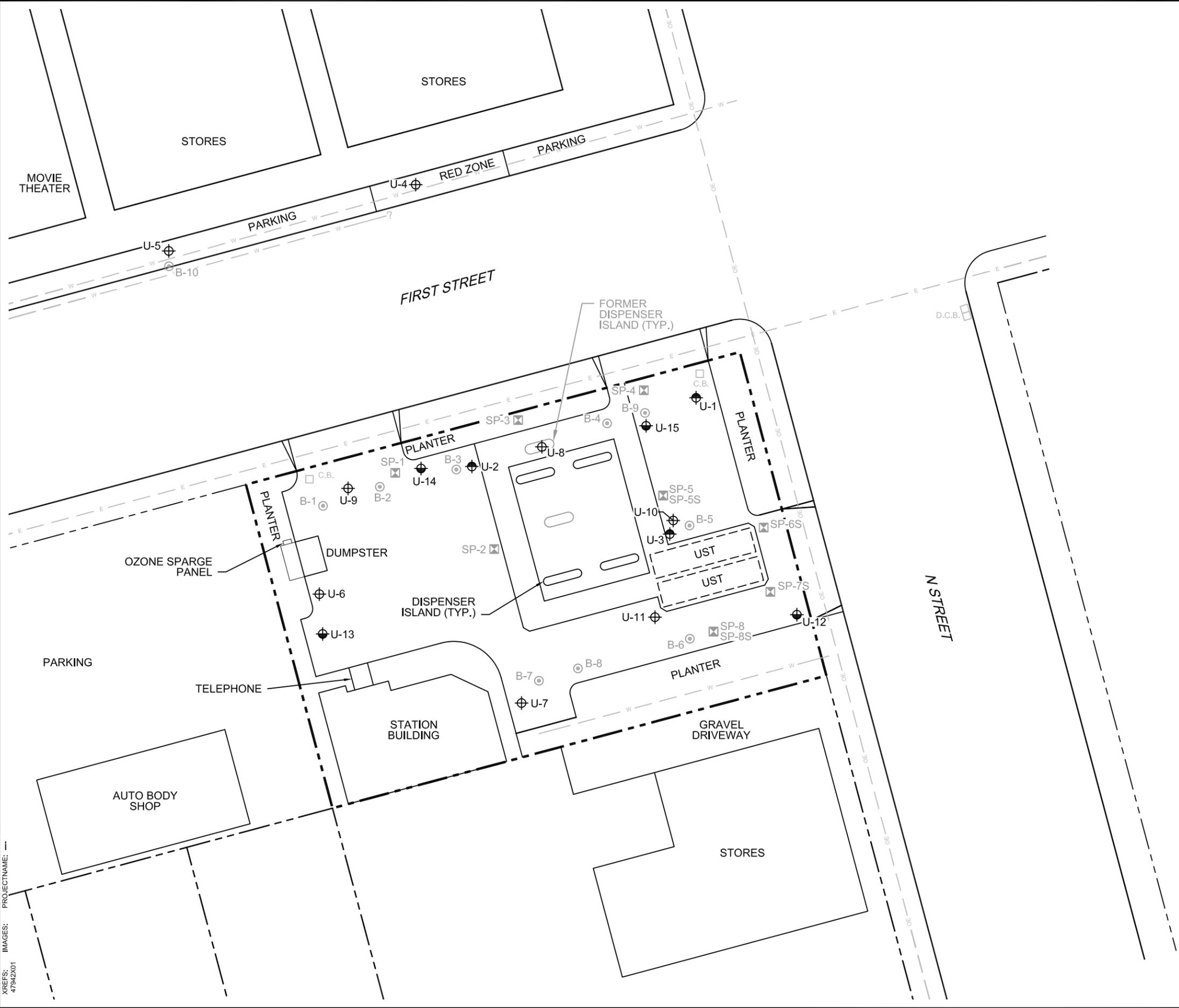
Somsamak, P., H.H. Richnow, and M.M. Häggblom. 2006. Carbon isotope fractionation during anaerobic degradation of methyl *tert*-butyl ether under sulfate-reducing and methanogenic conditions. *Applied and Environmental Microbiology* **72**(2): 1157-1163.

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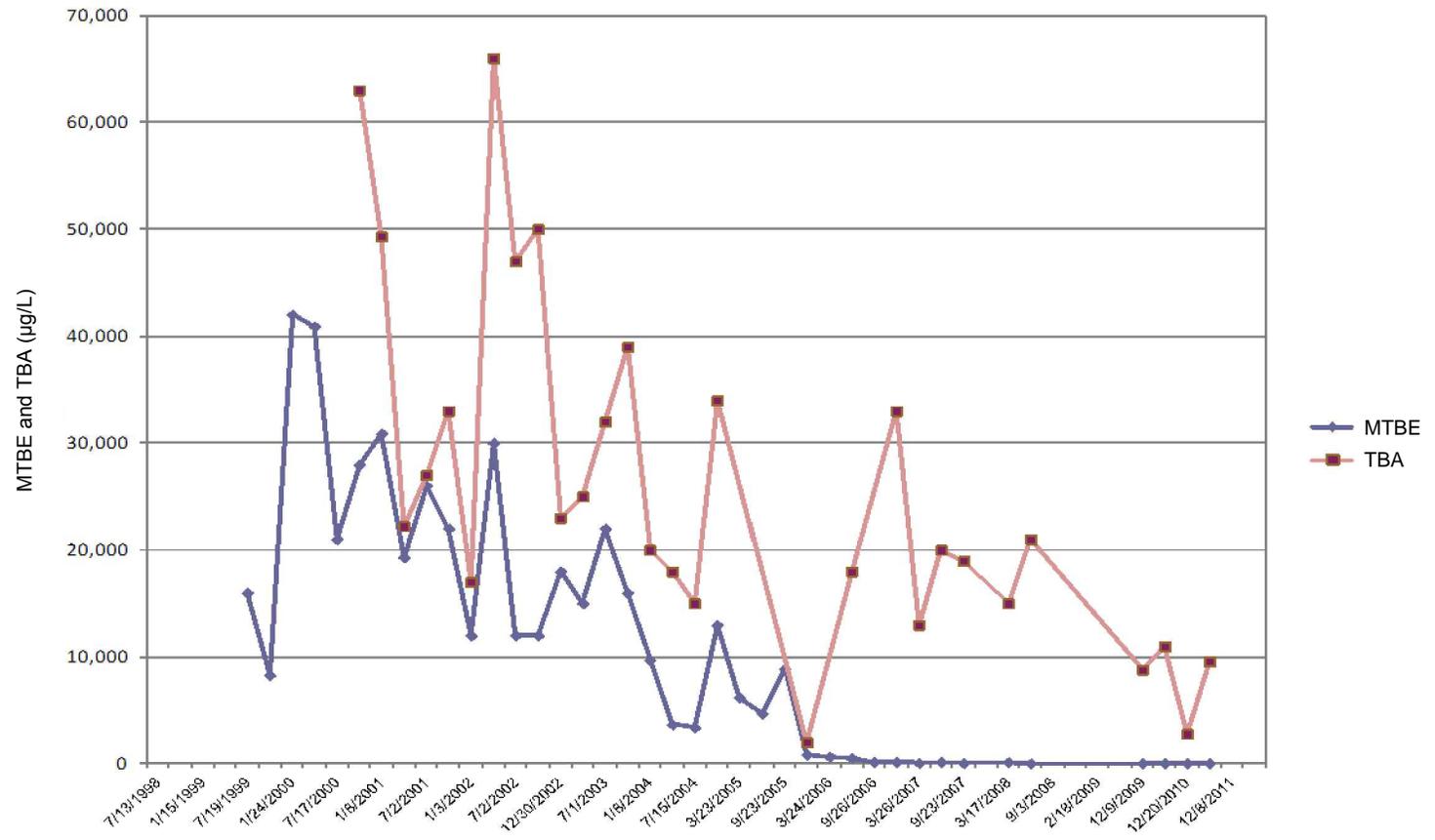
- LEGEND**
- PROPERTY BOUNDARY
 - U-1 ⊕ SHALLOW ZONE MONITORING WELL
 - U-6 ⊕ INTERMEDIATE ZONE MONITORING WELL
 - U-15 ⊕ DEEP ZONE MONITORING WELL
 - SP-1 ☒ OZONE SPARGE POINT
 - B-1 ⊙ BOREHOLE LOCATION
 - C.B. □ STORM DRAIN
 - - - UNDERGROUND ELECTRIC
 - - - UNDERGROUND WATER
 - - - OVERHEAD ELECTRIC

- NOTES:**
1. BASE MAP PROVIDED BY CRA, DATED 1/28/2011. BASED ON A MAP PROVIDED BY DELTA ENVIRONMENTAL CONSULTANTS, FIGURE 2, TITLED "SITE MAP", DATED 12/14/2005.
 2. ALL SITE FEATURES AND LOCATIONS ARE APPROXIMATE.



UNION OIL COMPANY OF CALIFORNIA 76 SERVICE STATION 4186 1771 FIRST STREET LIVERMORE, CALIFORNIA	
SITE PLAN	
	FIGURE 1

XREFS: IMAGES: PROJECTNAME: ----



NOTE: NO DETECTS ARE PLOTTED USING THE DETECTION LIMIT.

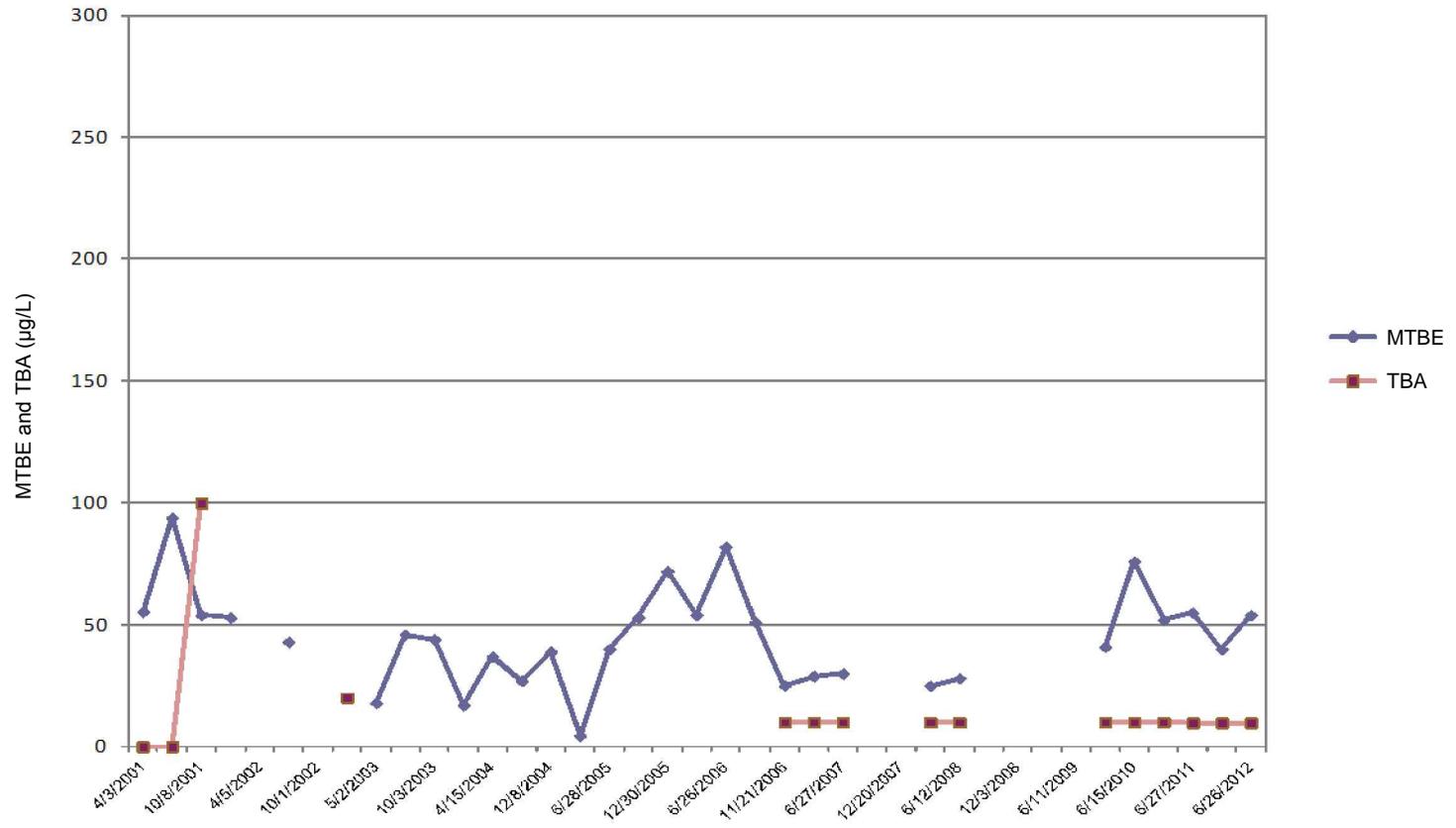
MTBE = METHYL TERTIARY BUTYL ETHER
 TBA = TERTIARY BUTANOL
 µg/L = MICROGRAMS PER LITER

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 LIVERMORE, CALIFORNIA

**MTBE AND TBA DATA
 WELL U-3**



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NOTE: NO DETECTS ARE PLOTTED USING THE DETECTION LIMIT.

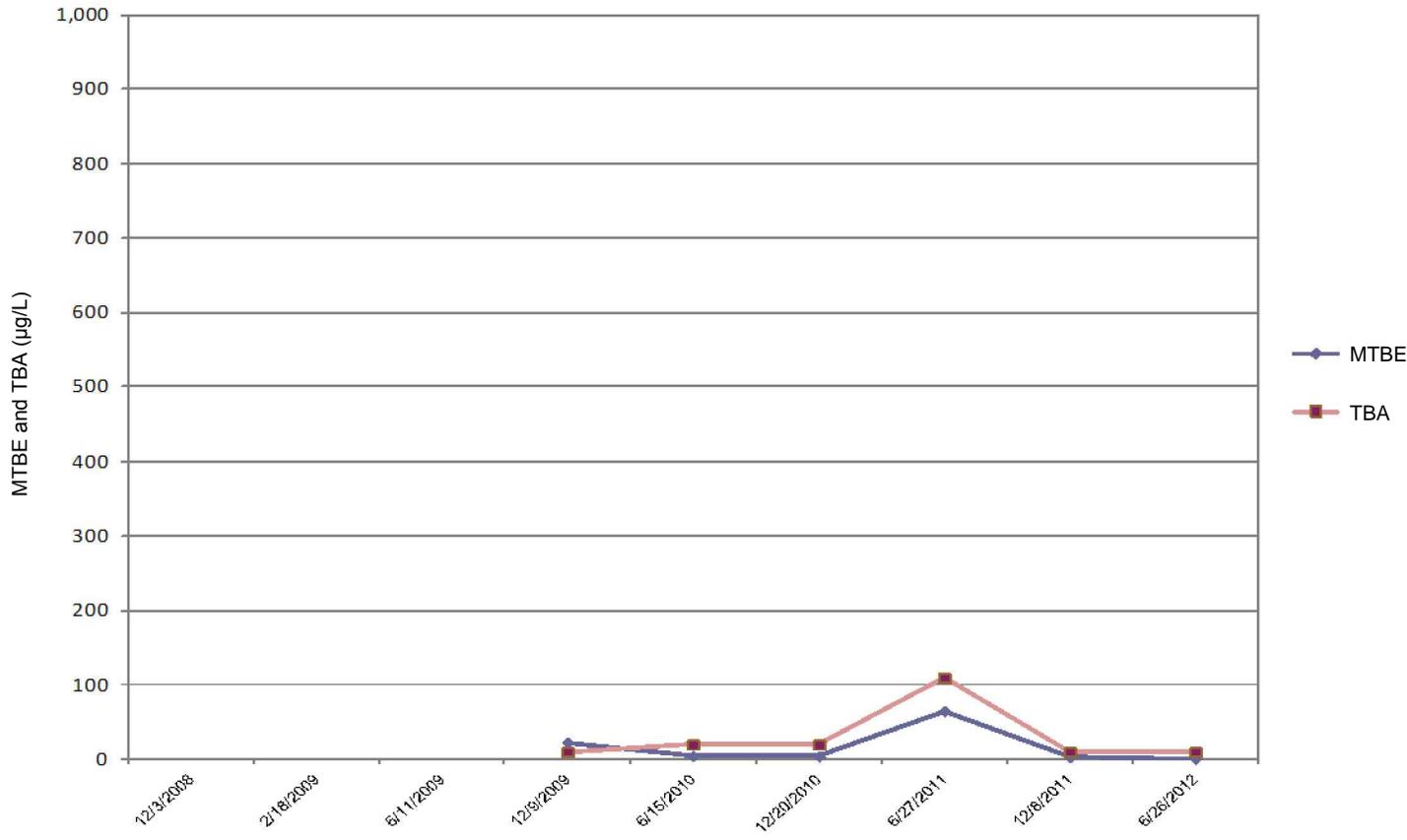
MTBE = METHYL TERTIARY BUTYL ETHER
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**MTBE AND TBA DATA
 WELL U-5**



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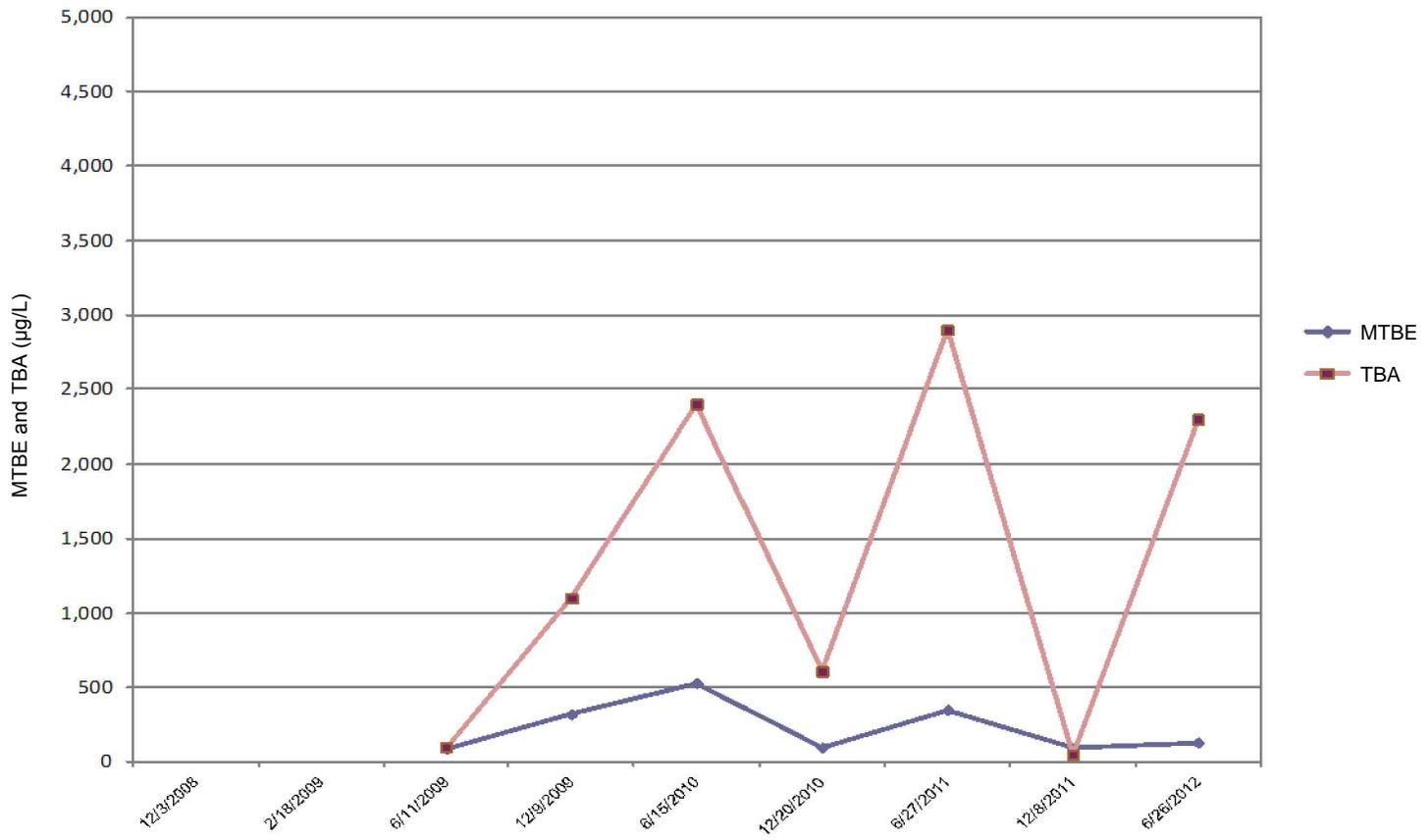
NOTE: NO DETECTS ARE PLOTTED USING THE DETECTION LIMIT.

MTBE = METHYL TERTIARY BUTYL ETHER
 TBA = TERTIARY BUTANOL
 µg/L = MICROGRAMS PER LITER

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**MTBE AND TBA DATA
 WELL U-9**

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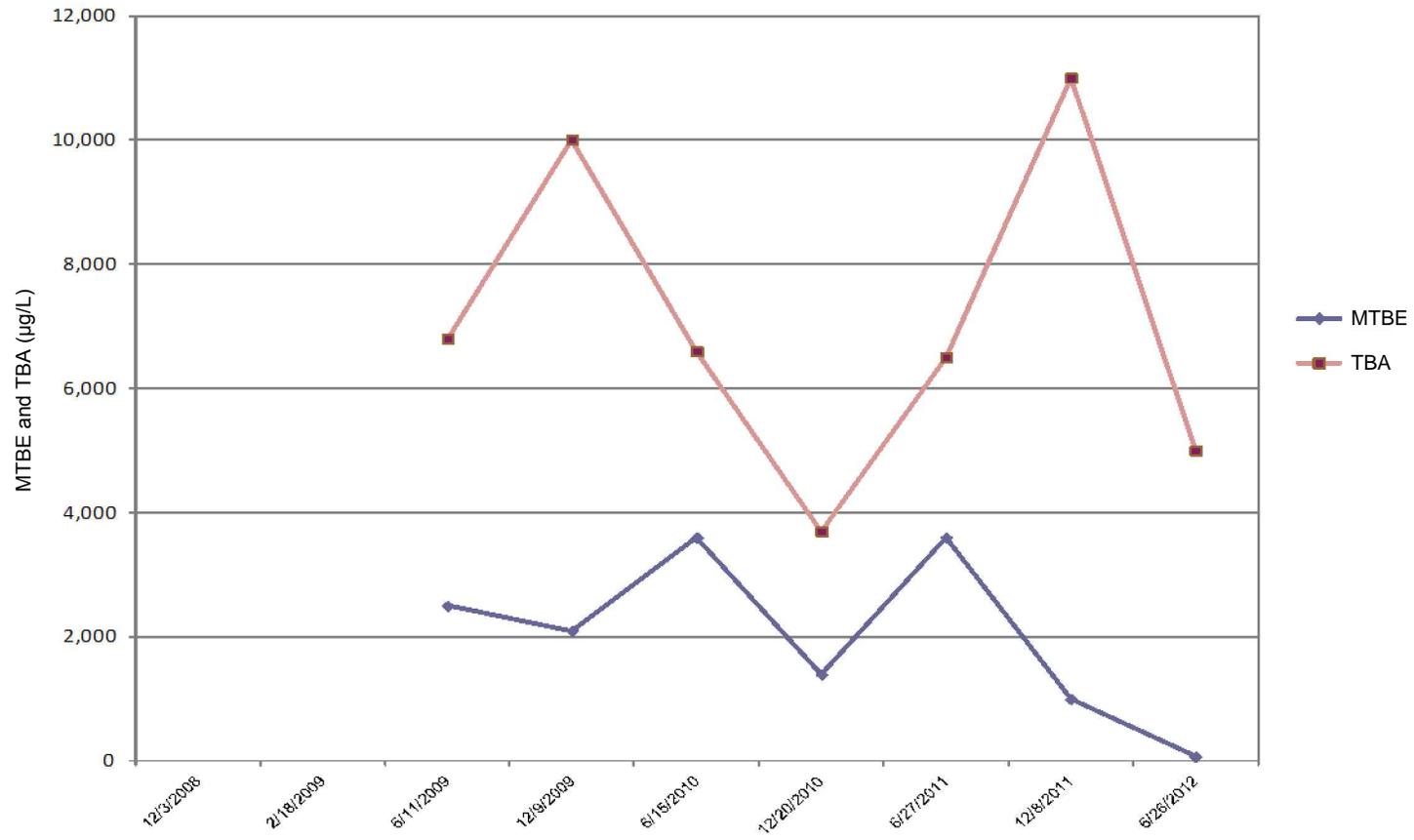
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MTBE = METHYL TERTIARY BUTYL ETHER
 TBA = TERTIARY BUTANOL
 µg/L = MICROGRAMS PER LITER

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**MTBE AND TBA DATA
 WELL U-10**

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NOTE: NO DETECTS ARE PLOTTED USING THE DETECTION LIMIT.

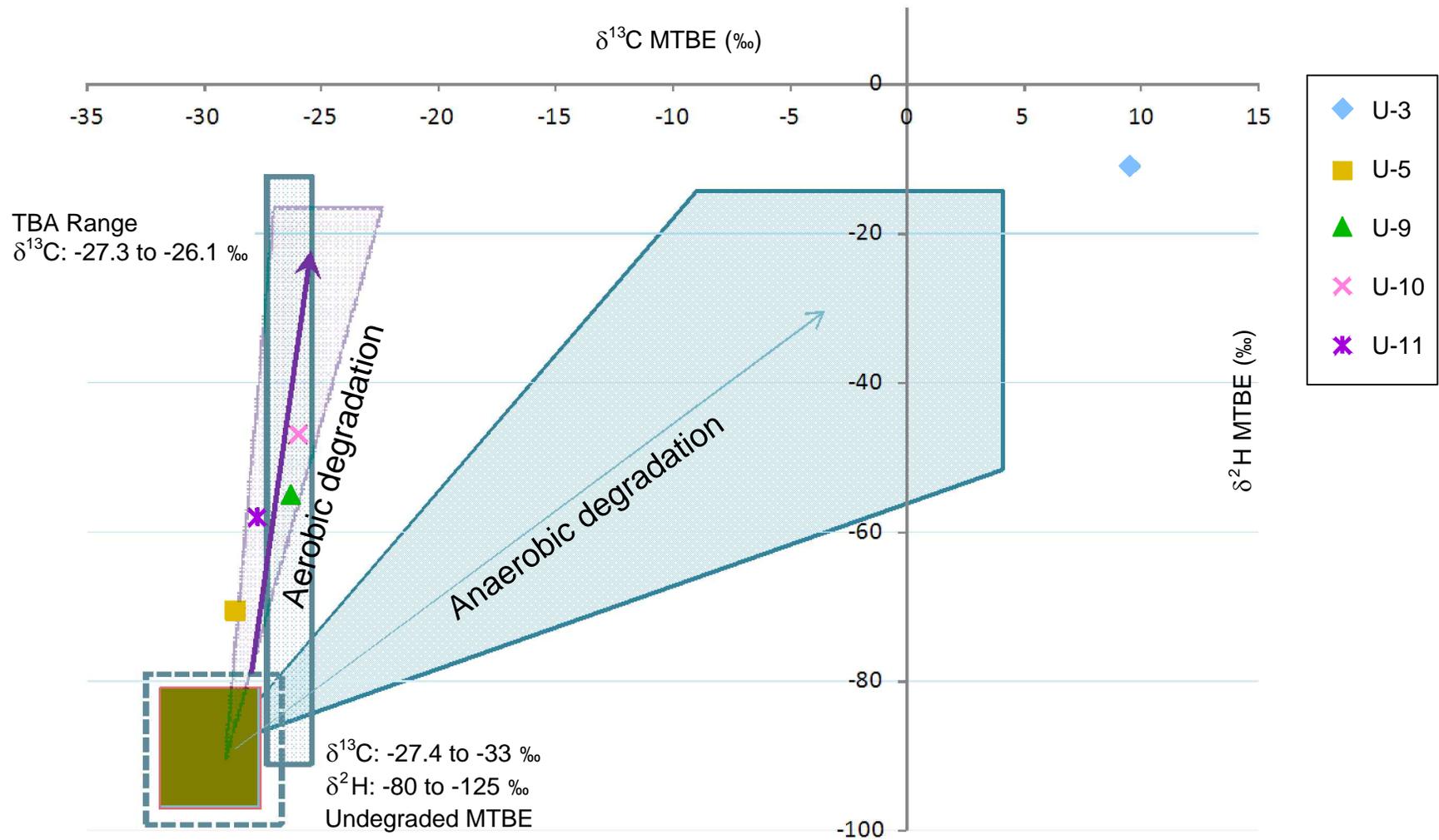
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TBA = TERTIARY BUTANOL
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**MTBE AND TBA DATA
WELL U-11**



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MTBE = METHYL TERTIARY BUTYL ETHER
 TBA = TERTIARY BUTANOL
 δ¹³C δ²H = ISOTOPIC SIGNATURE FOR CARBON AND HYDROGEN
 ‰ = PER MIL; PARTS PER THOUSAND

NOTE:
 Observed δ¹³C data range for TBA site data denoted as transparent narrow blue box. Anaerobic and Aerobic degradation trajectories are presented, as a result of the biodegradation of undegraded MTBE (the literature-derived data range is listed, as well as, graphically denoted with the yellow box, including error (the dashed border)).

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CARBON (δ¹³C) VERSUS HYDROGEN (δ²H) ISOTOPE VALUES FOR MTBE AT U-3, U-5, U-9, U-10, AND U-11

