

COMPARISON OF NAPHTHALENE AMBIENT AIR SAMPLING & ANALYSIS METHODS AT FORMER MANUFACTURED GAS PLANT (MGP) REMEDIATION SITES

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ABSTRACT

Naphthalene is a contaminant of concern at former Manufactured Gas Plant (MGP) and other property redevelopment sites across the country. A major component of coal tar waste and a possible human carcinogen (EPA Group C), naphthalene is a chemical that may adversely affect human health at remediation sites. Due to its boiling point and vapor pressure, naphthalene can exhibit both volatile and semi-volatile characteristics; therefore the question can arise as to how to properly measure naphthalene in ambient air.

Two commonly applied methods of measuring vapor phase naphthalene include EPA Method TO-15, which utilizes whole air sampling in passivated stainless steel canisters; and EPA Method TO-13A, which utilizes high volume sorbent based sampling with polyurethane foam/XAD resin cartridges. Analytical differences between these two methods will be discussed, keeping reference to naphthalene's unique chemical & physical properties.

This case study will present weekly data spanning a twelve month period (December 2006 – December 2007) from co-located EPA Method TO-15 and TO-13A ambient air samples at the perimeter of two MGP cleanup remediation sites. Distinct trends are noted and discussed in this paper when comparing the concentration results from the two methods.

Keywords: Naphthalene, TO-15, TO-13A, perimeter air monitoring, fenceline air monitoring

1. INTRODUCTION

As part of many former MGP site fenceline air monitoring programs conducted in association with site remediation tasks, volatile organic compounds (VOCs), particulates, and polycyclic aromatic hydrocarbon (PAH) compounds have been routinely monitored. Perimeter air quality monitoring is generally performed during site activities which may generate emissions associated with the investigation and remediation / dredging of the former MGP sites. The perimeter air monitoring program is generally designed to accomplish the objectives described below:

- Establish background levels of target analytes in ambient air;
- Develop action levels which are protective of public health for particulates (i.e. PAHs) and VOC vapors at the site;
- Monitor and document perimeter ambient air levels of target analytes during the days when site activities may produce air emissions; and
- Evaluate the need for dust and / or vapor control measures to reduce airborne compounds.

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All perimeter air monitoring systems (see Figure 1) are intended to be protective of public health in the vicinity of the remediation sites. The sampling program is typically designed to provide air monitoring during days of potential air emissions so that acceptable risks for acute and sub-chronic exposures are not exceeded at the potential fence line receptor locations.

Site owners generally perform ambient fence line monitoring during all ground intrusive portions of remedial programs. Compounds of concern that are typically associated with former MGP sites include semi-volatile organic compounds (including PAHs) and VOCs. Therefore, the perimeter air-monitoring programs are generally targeted for analytes associated with these compounds. More specifically, particulate matter smaller than 10 microns in diameter (PM₁₀) and VOCs are generally proposed as the target analytes to be included in any perimeter ambient air monitoring program. Measurement of PM₁₀ often serves two purposes: they may act as a surrogate for PAHs, plus monitoring for PM₁₀ helps to protect against fugitive particulate emissions.

A significant part of many fence line ambient air monitoring programs is the integrated air sampling which is generally conducted at upwind and downwind locations. Using EPA approved sampling and analytical methods, the integrated air sampling documents ambient levels of specific target contaminants. Analyses are performed by an accredited analytical laboratory demonstrating proficiency and state certification for the specific analytical methods.

Emissions of pollutants such as PAHs, the BTX (benzene, toluene, and xylenes) compounds, and more recently naphthalene have become significant health & safety issues associated with the clean up of former MGP sites. There has been an increase in awareness and concern in measuring the concentration of these parameters as well as various odor-related parameters from remediation sites. The need to prevent/reduce community issues associated with remedial efforts has necessitated this awareness and concern. In addition, the recent re-evaluation of naphthalene's toxicity by US EPA has further promoted this increased interest in measuring naphthalene at these sites.

1.1 Chemical Properties of Naphthalene

Naphthalene (CAS number 91-20-3) is the simplest PAH compound, consisting of two fused benzene rings. Naphthalene has the somewhat unique chemical property of existing as a solid at room temperature (boiling point of 218°C) but also sublimating easily at room temperature. Its strong, characteristic mothball odor has a fairly low odor detection threshold (typically ranging from 200 to 440 µg/m³)^{3,9}, making it a potential driver for odor complaints at MGP remediation sites.

In addition, due to its vapor pressure of 0.087 mm Hg at 25°C, naphthalene is sometimes considered by the analytical laboratory community to be a "borderline" volatile/semi-volatile compound, since it may often be detected in both traditional VOC and PAH analyses. Due to its vapor pressure and tendency to sublime, in ambient air, naphthalene is known to mainly exist in the vapor phase (as opposed to the aerosol or particulate phase)^{7,8}.

1.2 Naphthalene Measurement Techniques

The use of continuous analyzers for the detection of total volatile organic compounds (TVOC) and BTX parameters has been helpful in addressing most of the issues associated with the sampling for these constituents. However, due to the limited number and the complexity of continuous/real-time analyzers for naphthalene (i.e. field gas chromatographs (GCs), open path Fourier transform infrared spectroscopy (FTIR), GC/surface acoustic wave detection (GC/SAW (e.g. Z-nose)), etc.), several integrated sampling approaches have been traditionally used to quantify naphthalene levels along the perimeter of MGP site remediation. Some of the most common integrated sampling / analytical approaches are as follows:

- SUMMA canister sampler, followed by EPA TO-15 VOC lab analysis
- High volume PUF/XAD sampler, followed by EPA TO-13A PAH lab analysis

- Thermal desorption tube sampler, followed by EPA TO-17 lab analysis

EPA TO-15 defines a VOC as having a vapor pressure greater than 0.1 mm Hg at 25°C and 1 atm. Naphthalene falls just below that threshold with a vapor pressure of 0.087 mm Hg. However, despite its low vapor pressure, analytical laboratories have been able to reliably quantitate naphthalene in canisters via EPA TO-15. In 2007, Hayes et al. reported that naphthalene behaved similarly to toluene in terms of calibration precision and linearity, even at very low (sub-ppbV) concentrations, which would be similar to the concentrations in ambient air⁵. Additionally, a 2007 Columbia Analytical Services canister stability study using real world ambient air showed acceptable recovery of naphthalene from canisters even after 30 days⁴.

EPA TO-13A has several limitations noted in the text of the method itself, including a notation that the polyurethane foam (PUF) media alone (i.e. not in combination with XAD-2 resin) has a sampling efficiency of only approximately 35% for naphthalene. In addition, for naphthalene, PUF alone has a lower recovery efficiency and storage capability, and has a tendency for breakthrough at higher temperatures (e.g. summer)². Finally approximately one year after the EPA TO-13A method was published, one of the authors of the method (Dr. Robert Lewis, EPA) posted responses to questions and comments related to the method. In one of his responses, Dr. Lewis states “Note also that TO-13A is not good for naphthalene at all with PUF and only marginally with XAD”¹⁰.

EPA TO-17¹¹ allows great flexibility in terms of the sorbent material used in the tubes, the sampling flow rate/duration, etc. Hydrophobic solid sorbents such as Tenax (2,6-diphenylene-oxide polymer resin) are excellent for sampling heavier molecular weight compounds such as naphthalene; however, due to the nature of the sorbent materials, these tubes may not be optimal for sampling a long list of VOCs/SVOCs with different chemical properties (e.g. molecular weight, polarity, adsorptive properties, etc.). In addition, it is impractical to take long duration (i.e. 8-24 hour) samples with this style of tube due to breakthrough volume limitations, among other issues.

Therefore, for the sites presented in this case study, EPA TO-15 was chosen (to also capture the BTX compounds) and EPA TO-13A was chosen (to also capture the other PAH compounds) for the integrated air sampling portion of the air monitoring program.

This paper presents a side-by-side comparison of naphthalene measurements collected using two of the above integrated sampling / analysis techniques (EPA Methods TO-13A and TO-15). The study was conducted over a one-year period (December 2006 – December 2007) at two ENSR operated MGP Site fenceline Air Monitoring (AM) programs on the East coast of the United States.



Figure 1. Photograph of typical perimeter air monitoring station used in this case study.

2. MATERIALS AND METHODS

Integrated naphthalene sampling for the test sites was performed on a weekly routine basis following EPA TO-15 (VOC) and EPA TO-13A (PAH). At a minimum, three VOC and PAH samples, one upwind and two downwind for each method, were collected during each sampling day (usually 8-10 hours per day). The sampling locations were chosen from the various existing perimeter air monitoring locations and were based on actual and predicted wind conditions for the sampling day.

2.1 VOC Sampling: EPA Method TO-15

VOC samples were collected in pre-cleaned and batch certified 6L electropolished (passivated) stainless steel (i.e. SUMMA or equivalent) canisters in accordance with EPA Method TO-15¹. Please refer to Figure 2 for a photograph of the EPA TO-15 sampling.

Whole air samples were collected in evacuated stainless steel canisters (see Figure 3). Each sample was collected via a sampling cane from a height of approximately four feet, which was designed to approximate the typical breathing zone. The canister passively filled with sample air via a variable constant differential flow controller, which uses a critical orifice/diaphragm assembly to allow for uniform filling of the canister over the desired sampling period.

The VOC samples were analyzed by gas chromatography/mass spectrometry (GC/MS in full scan mode) for a 44 VOC compound list. The off-site analyzing laboratory maintained relevant laboratory certification from the National Environmental Laboratory Accrediting Conference (NELAC) and the governing state accreditation body.



Figure 2. Photograph of EPA TO-15 sampling with stainless steel canister.



Figure 3. Detailed photograph of stainless steel canister, analog vacuum gauge, and flow controller.

2.2 PAH Sampling: EPA Method TO-13A

PAH samples were collected on pre-cleaned and certified high volume cartridges filled with a combination of polyurethane foam and XAD-2 resin (PUF/XAD) in accordance with EPA Method TO-13A².

The high volume PUF/XAD sampler (see Figure 4) consists of a sample head inlet which contains the sampling media (see Figure 5), a high volume air blower which allows a large quantity of air to be drawn through the sampling media, and flow controllers and timers to quantify the sampling flow rates (generally around 6.4 standard cubic feet per minute (SCFM) or 180 standard liters per minute (SLPM)) and sample volumes (approximately 110-120 m³ over 8-10 hours). Sample air passes through the sample head and then through the PUF/XAD cartridge, where the vapor phase fraction of the semi-volatile compounds are adsorbed on the sampling media and the aerosol phase fraction of the semi-volatile compounds are collected physically on the PUF/XAD sampling media as well as an inline quartz fiber filter.

Prior to sample collection, each PUF/XAD sampler was calibrated using a calibration inlet according to TO-13A protocols. At the completion of each sample day, the PUF/XAD cartridge sample was removed from the sample head, wrapped in aluminum foil, labeled, and placed in a cool container (on ice) for shipment to the laboratory for analysis.

The PAH samples were then extracted using a soxhlet procedure, concentrated by evaporation, and the concentrated extract was analyzed by gas chromatography/mass spectrometry (GC/MS in Selected Ion Monitoring (SIM) mode) for an 18 PAH compound list. The off-site analyzing laboratory maintained relevant laboratory certification from the National Environmental Laboratory Accrediting Conference (NELAC) and the governing state accreditation body.

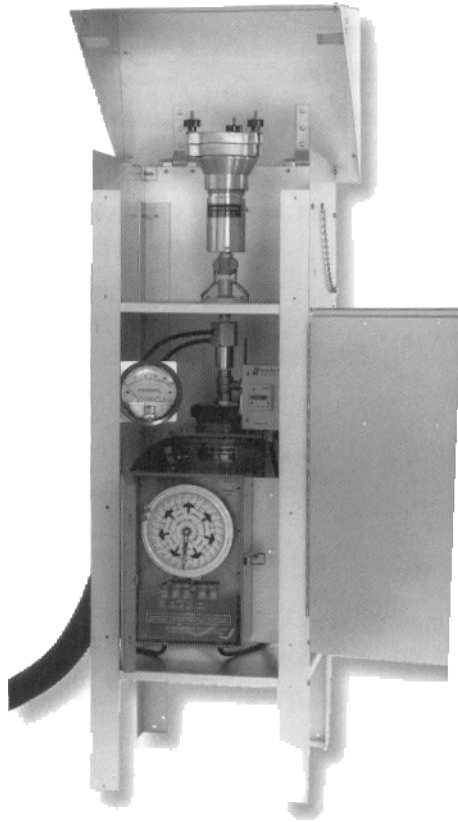


Figure 4. Photograph of EPA TO-13A high volume PUF/XAD sampler.



Figure 5. Detailed photograph of high volume PUF/XAD sampling media cartridge.

RESULTS AND DISCUSSION

From the co-located data collected between December 2006 and December 2007, a total of 105 paired data points (where naphthalene was positively detected via both analytical methods) were evaluated in this study (Table 1). For each set of paired data, the relative percent difference (RPD) between the naphthalene concentration reported from EPA TO-15 and the naphthalene concentration reported from EPA TO-13A was calculated, and the ratio of the EPA TO-15 concentration to the EPA TO-13A concentration was also calculated (i.e. EPA TO-15 concentration divided by EPA TO-13A concentration). The standard deviation of the ratios was calculated, along with the upper and lower control limits (equal to \pm three standard deviations).

The average EPA TO-15: EPA TO-13A ratio for all 105 data points was 4.01, meaning that on average, the EPA TO-15 concentrations seen for naphthalene were 4.01x higher than the EPA TO-13A concentrations seen for naphthalene. The average RPD was quite high at 97%, and similarly the standard deviation of the ratios was very high at 3.50. Of the 105 data points, there were three outlier data points with ratios over 10.51 (the upper control limit). If these three data points were removed from the data set, the average ratio changed to 3.61 and the standard deviation dropped to 2.55, demonstrating that there was still a sizable spread in the data. However, only nine out of the 105 ratios was less than one (meaning that the concentration of naphthalene seen from EPA TO-13A was greater than the concentration of naphthalene seen from EPA TO-15). Refer to Table 2 for a summary of relevant statistics and Figure 6 for a graphical representation of the ratios over time with upper and lower control limits.

Table 1. EPA TO-15 & TO-13A concentrations for 105 paired samples (* = Outliers)

Date Sampled	Concentration EPA TO-13A ($\mu\text{g}/\text{m}^3$)	Concentration EPA TO-15 ($\mu\text{g}/\text{m}^3$)	RPD	Ratio EPA TO-15 : TO-13A
11-Dec-06	0.28	1.40	133%	5.00
11-Dec-06	1.24	1.60	25%	1.29
11-Dec-06	0.10	0.89	159%	8.86
12-Dec-06	1.73	2.20	24%	1.27
12-Dec-06	0.07	0.68	163%	9.74
12-Dec-06	0.15	0.70	129%	4.65
13-Dec-06	1.33	2.10	45%	1.58
13-Dec-06	0.67	0.98	38%	1.47
13-Dec-06	0.67	0.98	38%	1.47
14-Dec-06	0.23	1.30	140%	5.65
14-Dec-06	0.90	2.50	94%	2.78
14-Dec-06	0.11	0.84	154%	7.66
15-Dec-06	1.89	3.20	51%	1.69
15-Dec-06	0.12	0.78	146%	6.46
19-Dec-06	0.46	0.36	23%	0.79
27-Dec-06	0.14	0.94	148%	6.69
4-Jan-07	0.98	1.90	64%	1.94
4-Jan-07	0.15	0.56	115%	3.70
9-Jan-07	1.17	2.70	79%	2.31
9-Jan-07	0.16	0.54	109%	3.40
18-Jan-07	2.24	3.10	32%	1.38
18-Jan-07	0.82	1.20	38%	1.46
18-Jan-07	0.17	0.48	95%	2.82
24-Jan-07	0.12	0.33	93%	2.75
24-Jan-07	0.06	0.34	140%	5.65
24-Jan-07	0.07	0.37	136%	5.23

Date Sampled	Concentration EPA TO-13A ($\mu\text{g}/\text{m}^3$)	Concentration EPA TO-15 ($\mu\text{g}/\text{m}^3$)	RPD	Ratio EPA TO-15 : TO-13A
31-Jan-07	1.32	2.60	65%	1.97
31-Jan-07	0.07	0.42	143%	5.98
22-Feb-07	1.67	4.00	82%	2.40
22-Feb-07	0.57	1.80	104%	3.16
22-Feb-07	2.07	14.00	148%	6.76
1-Mar-07	1.12	2.50	76%	2.23
1-Mar-07	0.12	1.30	166%	10.83
1-Mar-07	0.09	0.73	156%	8.09
7-Mar-07	2.24	3.20	35%	1.43
7-Mar-07	0.11	0.47	124%	4.23
7-Mar-07	0.19	0.78	122%	4.12
13-Mar-07	0.70	2.20	103%	3.14
13-Mar-07	0.29	1.40	131%	4.83
13-Mar-07	2.26	1.03	75%	0.46
21-Mar-07	1.98	1.40	34%	0.71
29-Mar-07	1.35	1.30	4%	0.96
29-Mar-07	0.78	4.40	140%	5.64
29-Mar-07	0.04	0.59	175%	14.73
4-Apr-07	0.81	2.60	105%	3.21
4-Apr-07	0.37	2.40	147%	6.49
4-Apr-07	0.06	0.87	174%	14.56
10-Apr-07	11.27	14.00	22%	1.24
10-Apr-07	0.24	0.55	79%	2.29
19-Apr-07	0.60	1.30	74%	2.17
19-Apr-07	3.13	1.40	76%	0.45
19-Apr-07	0.09	0.70	154%	7.77
26-Apr-07	0.99	2.20	76%	2.22
26-Apr-07	0.06	1.40	184%	23.33
26-Apr-07	0.10	0.59	142%	5.94
1-May-07	1.05	1.50	35%	1.43
1-May-07	7.75	3.90	66%	0.50
1-May-07	0.11	0.88	155%	7.96
7-May-07	0.55	1.40	87%	2.55
7-May-07	0.08	0.68	158%	8.56
7-May-07	0.14	0.88	145%	6.32
15-May-07	1.79	7.30	121%	4.08
15-May-07	0.14	0.87	145%	6.22
15-May-07	0.07	0.82	168%	11.66
23-May-07	0.12	0.64	137%	5.33
23-May-07	0.52	1.20	79%	2.31
23-May-07	0.24	0.92	117%	3.83
23-May-07	3.30	0.27	170%	0.08
30-May-07	0.06	0.24	121%	4.08
30-May-07	0.05	0.25	134%	5.07
30-May-07	0.06	0.52	158%	8.62
7-Jun-07	0.06	0.43	151%	7.20
7-Jun-07	0.07	0.26	115%	3.70
14-Jun-07	0.05	0.21	123%	4.22
27-Jun-07	0.33	0.67	68%	2.03
5-Jul-07	0.14	0.42	100%	2.98
11-Jul-07	0.17	0.30	56%	1.78
11-Jul-07	0.14	0.56	119%	3.97
17-Jul-07	0.09	0.30	108%	3.36

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Date Sampled	Concentration EPA TO-13A ($\mu\text{g}/\text{m}^3$)	Concentration EPA TO-15 ($\mu\text{g}/\text{m}^3$)	RPD	Ratio EPA TO-15 : TO-13A
17-Jul-07	0.36	0.52	36%	1.44
26-Jul-07	0.27	0.73	92%	2.72
2-Aug-07	0.50	0.95	62%	1.90
2-Aug-07	0.06	0.29	132%	4.86
7-Aug-07	0.09	0.23	88%	2.56
7-Aug-07	0.21	0.63	100%	3.00
7-Aug-07	0.07	0.25	113%	3.58
15-Nov-07	0.17	0.25	36%	1.44
15-Nov-07	0.05	0.20	121%	4.08
15-Nov-07	0.07	0.43	144%	6.13
20-Nov-07	0.23	0.31	30%	1.35
20-Nov-07	0.26	0.22	19%	0.83
29-Nov-07	0.30	2.30	154%	7.67
29-Nov-07	1.70	2.60	42%	1.53
29-Nov-07	0.10	0.41	122%	4.13
3-Dec-07	0.37	0.80	74%	2.16
3-Dec-07	0.32	0.41	24%	1.27
11-Dec-07	0.61	0.68	11%	1.11
11-Dec-07	1.28	2.60	68%	2.03
11-Dec-07	2.18	0.91	82%	0.42
19-Dec-07	1.09	2.00	59%	1.83
19-Dec-07	0.24	0.32	30%	1.35
19-Dec-07	0.10	0.23	79%	2.31
27-Dec-07	1.69	2.30	31%	1.36
27-Dec-07	2.74	4.50	49%	1.64
27-Dec-07	0.28	0.29	5%	1.05

Table 2. Summary of Relevant Statistics

Average Ratio (EPA TO-15 : EPA TO-13A), n=105	4.01
Average Ratio (EPA TO-15 : EPA TO-13A), no outliers, n=102	3.61
Standard Deviation of Ratios n=105	3.50
Standard Deviation of Ratios, no outliers n= 102	2.55
Percent of Paired Samples where EPA TO-15 concentration > EPA TO-13A concentration, n=105	91.4%
Percent of Paired Samples where EPA TO-13A concentration > EPA TO-15 concentration, n=105	8.6%

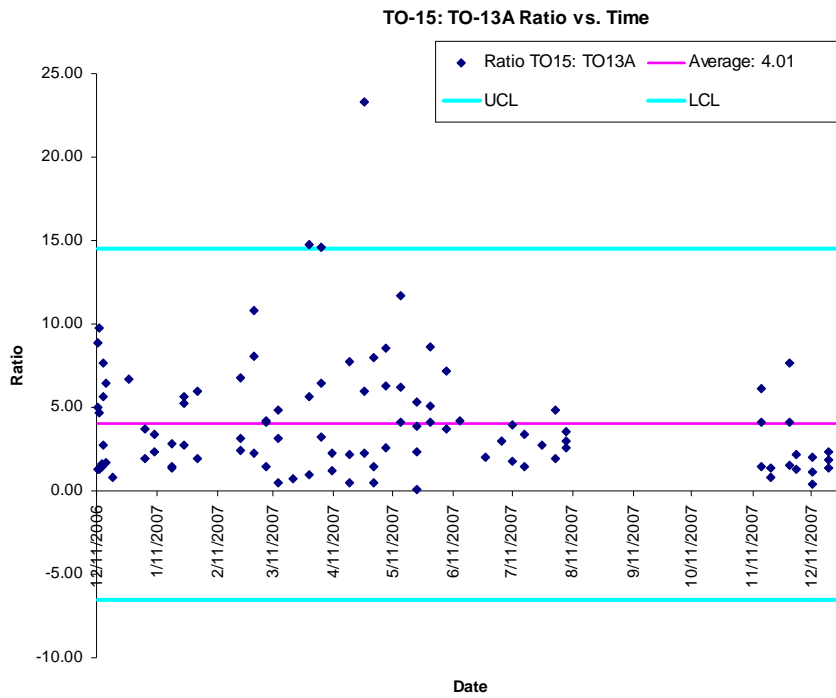


Figure 6. EPA TO-15 : EPA TO-13A ratio over time, showing upper and lower control limits

In general, the EPA TO-15 and EPA TO-13A concentration trends paired well, such that when relative higher concentrations of naphthalene were seen in one method, relative higher concentrations were also seen in the other method. Refer to Figure 7 for a graph showing both sets of concentrations over time.

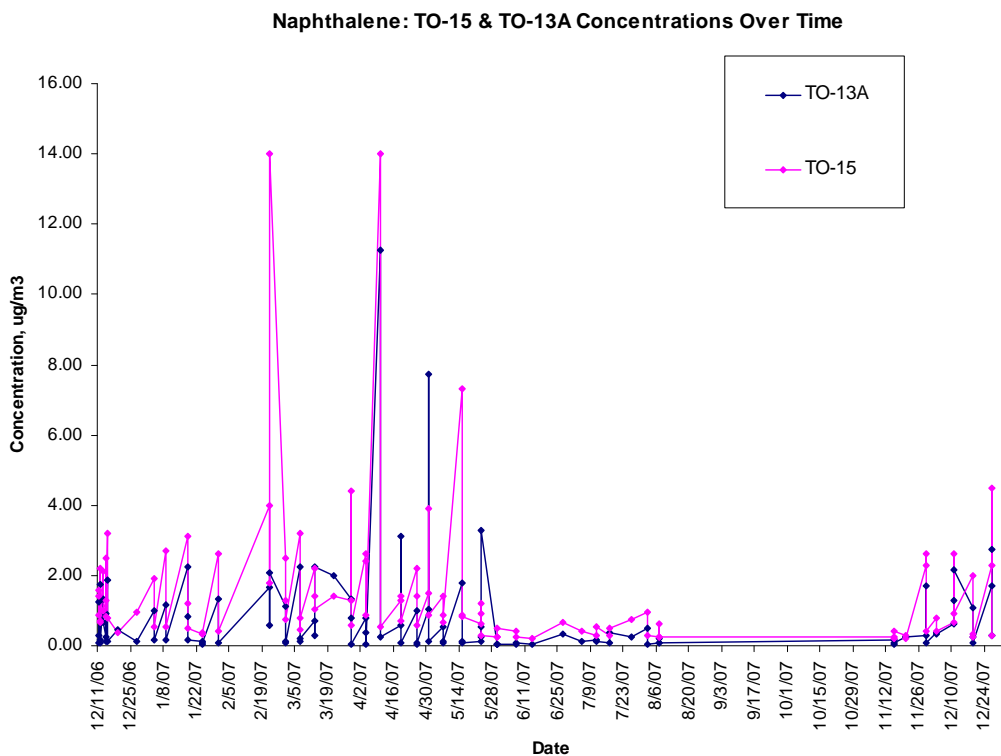


Figure 7. EPA TO-15 and EPA TO-13A concentrations over time, showing similar trend patterns

When the concentration of naphthalene from EPA TO-15 was plotted against the concentration of naphthalene from EPA TO-13A, poor linearity ($R^2=0.4835$) was observed, implying that there was no direct relationship between the two sets of data from the two different sampling/analytical methods (Figure 8).

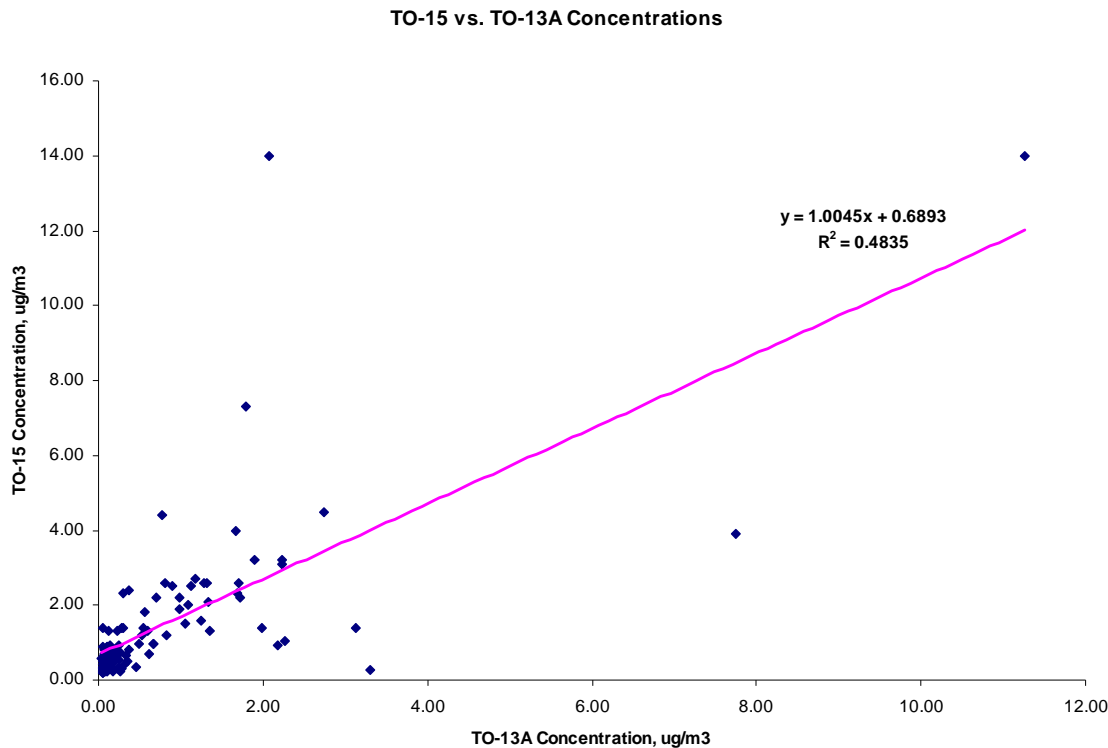


Figure 8. EPA TO-15 vs. EPA TO-13A concentrations, showing poor linearity

CONCLUSION

Results from this study show that the EPA TO-15 sampling/analytical method in general yields a higher concentration result for vapor phase naphthalene than EPA TO-13A. Similar results from a MGP perimeter monitoring case study were presented at the 2006 Natural Gas Technologies (GTI) Conference in Orlando, FL⁶. No discernable trends were noted related to sampling date (and therefore average ambient temperature), sampling location, or naphthalene concentration level.

There are a few analytical facts that may contribute to the observed EPA TO-15 concentrations being higher than the observed EPA TO-13A concentrations. First, PUF and XAD-2 are both known to have marginal collection efficiency for vapor phase naphthalene. In addition, there is a potential for substantial losses of naphthalene (due to its tendency to sublime and its relatively high vapor pressure as compared to other PAHs) during EPA TO-13A soxhlet extraction & evaporative concentration.

When designing a perimeter ambient air monitoring program (that includes naphthalene) for MGP remediation sites, it is important to keep in mind the sampling/analytical method characteristics listed in Table 3. Since each site is different (and may have different data quality objectives), each air monitoring program should weigh the pros and cons of all analytical methods (both field and laboratory based) available before developing a work plan.

Table 3. Method comparison for EPA TO-15 and EPA TO-13A

EPA TO-15	EPA TO-13A
Reporting limits: 0.2-0.5 ug/m ³	Reporting limits: 0.01-0.03 ug/m ³
Other VOC data available (BTX, etc.)	No VOC data available
No other PAH data available	Other PAH data available
No additional sampling equipment needed	High volume air sampler needed
No sample preservation needed	Samples must be shipped cold to lab

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